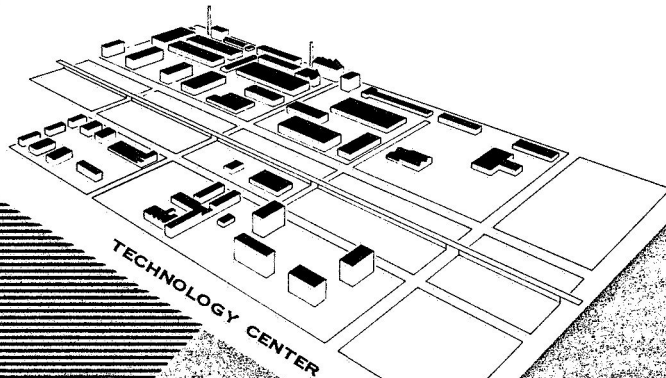


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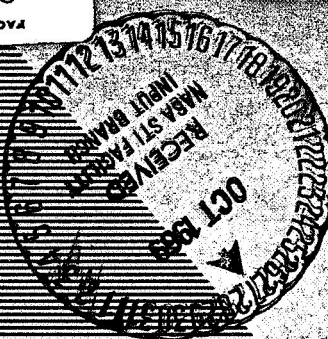
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ARF Project K60³05

HAZARD POTENTIAL OF DIFFUSION PUMP FLUID

Final Report

Period Covered - January 15, 1963 to June 15, 1963
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ARF Final Report
K6035

Final Report K6035
HAZARD POTENTIAL OF DIFFUSION
PUMP FLUID
by
C. W. Solbrig
W. E. Jamison

FOREWORD

This is the final report on Armour Research Foundation Project No. K6035 entitled "Hazard Potential of Diffusion Pump Fluid" and it covers the work performed on this project during the period of January 15, 1963 to June 15, 1963. This research was sponsored by the National Aeronautics and Space Administration, Manned Spacecraft Center in Houston, Texas.

The following Armour Research Foundation personnel have made useful contributions to this project: R. J. Logan, W. E. Jamison, T. Sytko, P. W. Cooper, R. Kamo, F. Iwatsuki and C. Solbrig.

All pertinent information and data are recorded in Armour Research Foundation Logbooks C13140, C13148, and C13506 and their appendices.

Respectfully submitted,

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ABSTRACT

HAZARD POTENTIAL OF DIFFUSION PUMP FLUID

Evaluation of the combustion hazard potential of five diffusion pump oils, specifically DC705, Convalex 10, DC-704, TCP and Convoil 20, was the objective of this investigation. Combustion tests conducted in a stainless steel cell for selection purposes, testified that DC 705 is the least hazardous of the oils. Detailed experiments in a glass cell indicated that DC 705 does not explode in the presence of any concentration of oxygen in the pressure range below 450 torr and temperature range below 600°F. Spark ignition experiments produced only one explosion below 700°F. Experiments conducted to determine the possibility of exothermic decomposition attested that detonation of any of these oils is not possible. The catalytic effect of stainless steel, steel, copper, and aluminum upon the hazard of DC 705 and Convalex 10 in pure oxygen was observed for a period of fifteen days during which no definite explosions were observed. The results of these bench tests substantiate the conclusion that these oils introduce no combustion hazard in a diffusion pump.

To confirm these results, oxygen was introduced up to 8 psia into two diffusion pumps, a 35 inch and a six inch, after two weeks of operation. The oils subjected to this test in the 35 inch pump were DC 704, and DC 705; in the six inch pump, DC 704, DC 705, and Convalex 10. In addition, energy was dissipated in the form of a spark in the chamber, barrel, and foreline of the six inch pump. As expected, no explosions occurred. Measurements detected no measurable backstreaming occurring in the 35 inch diffusion pump during 5.25 hours of operation for either DC 704 or DC 705.

As a result of this investigation, use of DC 705 is recommended.

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INTRODUCTION

In the course of the design of a Space Environmental Facility to be installed at Clear Lake, Texas by the National Aeronautics and Space Administration, concern over the explosion hazard potential of diffusion pump oils was expressed. Danger of explosion of the diffusion pump oils exists if a high pressure oxygen supply line is ruptured or if the chamber must be repressurized with oxygen to 5 psia because of a space suit failure. Excessive backstreaming of diffusion pump fluids can also create a hazard if the accumulation becomes sufficiently large.

Therefore, at the request of NASA, Armour Research Foundation began a program which had as its main objective, the determination of the hazard potential of diffusion pump oils. The program was divided into two phases: 1) the combustion study and 2) the high vacuum study. These phases were run concurrently with appropriate schedules to permit use of information from the combustion study in the high vacuum study. The objectives of the combustion study were:

- 1) Determination of the spontaneous ignition states of five diffusion pump oils in an atmosphere of oxygen as a function of pressure, temperature, and reactant concentration within specified limits.

- 2) Determination of the amount of electrical energy required by a spark to cause an explosion of the diffusion pump oils in an oxygen atmosphere as a function of chamber pressure and reactant concentration.

- 3) Determination of the catalytic effect of aluminum, copper, steel, and stainless steel upon the spontaneous ignition states of the diffusion pump oils.

The objectives of the high vacuum study were:

- 1) Determination of the probability of combustion of various diffusion pump fluids in a simulated-space environmental chamber with a high throughput of pure oxygen.

- 2) Determination of the approximate concentration of oil which can be expected to accumulate in a space environmental chamber as a result of backstreaming from a 35-inch pump past a chevron baffle with and without water cooling.

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3) Determination of the occurrence or non-occurrence of combustion of various diffusion pump fluids under preselected conditions in a simulated-space environmental chamber with an external ignition source at a location in the chamber, in the diffusion pump, and in the foreline.

The results of both phases were used to draw conclusions as to the hazard potential of each of the diffusion pump oils. If a hazard existed, methods of inhibition or suppression of the explosion hazard were to be suggested.

Because NASA desired this information as soon as possible, a program with a very tight time schedule was set up to finish the program 1.5 months before the contractual completion date. Several difficult problems arose which caused this schedule to be delayed but the program was completed one month ahead of schedule.

II CONCLUSIONS

1. Combustion experiments were conducted with five diffusion pump oils; DC-705, Convalex 10, DC-704, TCP and Convoil 20, in the stainless steel combustion apparatus. Based upon the results of these experiments and upon some additional information, DC-705 was selected as the least hazardous and best diffusion pump oil, although none of these oils were evaluated as hazardous in the conditions encountered in diffusion pumps.

2. To substantiate our opinion that DC-705 was a very good diffusion pump oil, very comprehensive experiments were conducted in the glass apparatus. No spontaneous ignitions were observed in the range of temperatures and pressures investigated. It is our opinion that DC-705 will not react with oxygen if the total pressure of the gas mixture is below 450 torr and the temperature is below 600° F if no external ignition sources are present.

3. Spark ignition experiments on DC-705 were run in conjunction with the experiments in the glass system. Only one ignition was observed below 700° F

4. Detonation experiments were conducted on all five oils and no detonations were observed.

5. Eight fifteen day catalytic experiments were conducted on Convalex 10 and DC-705 with four different metals: stainless steel, copper, steel, and aluminum in combustion cells. Pressure rises were observed in two of these tests which were probably due to leaks into the system. No definitive conclusion can be drawn about the effect of catalysts in general upon these reactions since the experiments were very limited in number and scope.

6. Each of the oils, DC-704, DC-705, and Convalex 10, were run in a six inch diffusion pump for a period of two weeks. At the end of the first run, oxygen was evacuated through the mechanical pump. This produced an explosion in the exit of the mechanical pump due to the mechanical oil.

7. The oils, DC-704 and DC-705, were run in the 35 inch diffusion pump. At the end of two weeks, oxygen was admitted to 8psia. No explosion occurred, again reinforcing conclusion one.

8. A technique of measuring backstreaming was developed and used in the 35 inch diffusion pump. We could detect no backstreaming for exposures up to 5 1/4 hours of operation for either DC-704 or DC-705. Only in one

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case was it observed and this point is in question because of improper pump shut down.

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III

RECOMMENDATIONS FOR FUTURE WORK

DC-705 has been shown experimentally to be an extremely stable oil in spite of the fact that equilibrium considerations indicate that it should be extremely explosive. This means that certain metals may be extremely dangerous in a DC-705-oxygen atmosphere because these metals may be able to unleash the potential energy of a reaction by increasing the reaction rate. The catalytic investigation in this work is not satisfactory to show that no catalytic effect could be demonstrated by other metals than those investigated or for that matter, these metals under different conditions. A two week run is not particularly meaningful as related to evaluation of catalysts. An experiment performed with the catalyst in the glass combustion cell would have been as satisfactory. A two week run can be used however to measure the amount of oil which is decomposing. Since a large molecule such as DC-705 must decompose before it reacts, the decomposition rate indicates a period of time in which these decomposition products could become hazardous.

In the course of this work, a method for measuring backstreaming was developed which is unique and quite useful. Since TCP is to be used in mechanical pumps which are connected directly to the chamber, backstreaming measurements should be made on TCP.

The discussion of the catalytic tests indicates that a combustion cell with DC-705 had been contaminated by some vacuum hose which had melted. Apparently a few days after the catalytic tests has been terminated, a large reaction occurred. (The heater had been left on.) This points out that possibly once DC-705 becomes contaminated, it becomes very hazardous. Perhaps this should be investigated. Practically speaking, the polymer material used for seals in the space chamber and in space suits may provide this contamination.

The explosion curves (or reaction rates) should be determined for DC-705 in order to be cognizant of limits to which the space chamber may be subjected during unusual testing.

Since TCP is to be used in the mechanical roughing and backing pumps, it would be very useful to determine explosion limit curves for this oil also.

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IV. THEORY

Explosion limits of combustible mixtures may be calculated if sufficient information is available. This information was found lacking in the literature for the combustibles under consideration.

It is possible to estimate the equilibrium concentration of a reaction and an estimation of the equilibrium of Convalex 10 is included in this section. The results of this calculation indicate that a large potential exists for completion of these reactions. After it has been shown that a potential exists, the mechanism of this reaction must be determined. With this thought in mind, theories of combustion are reviewed briefly and this review indicates that spontaneous ignition temperatures depend principally on reaction rates.

Reaction rates are dependent upon pressure, temperature, concentration, and catalysts. The method by which ignition by an external spark causes an explosion is reviewed. The fact, that a catalyst can increase the reaction rate and thus cause an explosion when large reaction potentials exist, is emphasized.

A. Calculation of Reaction Potentials

A chemical reaction which takes place in a very short interval of time is usually called an explosion. Calculation of the equilibrium concentration of a reaction will indicate the probability of a reaction.

The equilibrium constant of a reaction is related to the change in the standard state free energy of a reaction by the equation

$$-\Delta F^{\circ} = RT \ln K \quad (1)$$

where ΔF° is the change in the standard state free energy, R is the universal gas constant, T is the absolute temperature of the reaction at standard state conditions and K is the equilibrium constant of the reaction. In order to calculate the equilibrium concentration, it is necessary to calculate K.

The quantity, ΔF° , may be obtained from the literature in two ways. Either ΔF° may be tabulated or the relation

$$\Delta F^\circ = \Delta H^\circ - T \Delta S^\circ \quad (2)$$

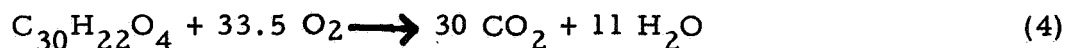
may be used with values of ΔH° , change in standard state enthalpy, and ΔS° , change in standard state entropy. The quantity $-\Delta H^\circ$ is generally referred to as the heat of reaction.

The quantity, ΔF° , may be calculated for a reaction from the free energies of formation of the components, ΔF_{fi} , involved in the reaction by the equation:

$$\Delta F^\circ = \sum_{i=1}^n a_i \Delta F_{fi} \quad (3)$$

where a_i are the stoichiometric coefficients in the reaction.

Consider the following equation for the reaction of Convalex 10:



From equation 3, we have:

$$\begin{aligned} \Delta F^\circ_{T_1} = & 30 \Delta F_{f CO_2 T_1} + 11 \Delta F_{f H_2O T_1} - (\Delta F_{f C_{30}H_{22}O_4 T_1} \\ & + 33.5 \Delta F_{f O_2 T_1}) \end{aligned} \quad (5)$$

where T_1 is usually taken at $25^\circ C$.

The quantity, ΔF_{fi} , is given in the literature for CO_2 , H_2O , and O_2 however the value of $\Delta F_{f C_{30}H_{22}O_4 T_1}$ is not and must be estimated. Using the method of Franklin⁽¹⁾, we have the result that $\Delta F_{f C_{30}H_{22}O_4 T = 25^\circ C} = 81.4 \text{ kcal/gmole}$. The procedure involved in the estimation of this free energy of formation is indicated in Table 1.

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TABLE 1
CALCULATION OF ΔF_{fi} $C_{30}H_{22}O_4$ $T = 25^\circ C$

Group	Contribution (i)	Number of Groups	Σi
$\begin{array}{c} \nearrow \\ \nwarrow \end{array} C-H$	4.84	22	106.5
$\begin{array}{c} \nearrow \\ \nwarrow \end{array} C-$	8.76	8	70.1
-O-	-23.8	4	-95.2
			$\Sigma(\Sigma i)$ 81.4 kcal/gmole

The same type of formulas may be applied to the calculation of ΔH° . The estimation of $\Delta H_f C_{30}H_{22}O_4$ $T = 25^\circ C$ is shown in Table 2.

TABLE 2
CALCULATION OF ΔH_f $C_{30}H_{22}O_4$ $T = 25^\circ C$

Group	Contribution (i)	Number of Groups	Σi
C-H	3.30	22	72.6
c-	5.57	8	44.6
-O-	-27.2	4	-108.8
			$\Sigma(\Sigma i)$ 8.4 kcal/gmole

Free energy and enthalpy obtained from Smith and Van Ness⁽²⁾ for CO_2 and H_2O are shown in Table 3.

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TABLE 3
HEATS AND FREE ENERGIES OF FORMATION

Substance	$\Delta H_{fi} \text{ T} = 25^\circ\text{C}$	$\Delta F_{fi} \text{ T} = 25^\circ\text{C}$
$\text{CO}_2(\text{g})$	-94.052	-94.260
$\text{H}_2\text{O}(\text{g})$	-57.798	-54.635

The heats and free energies of formation are defined to be zero for elements such as O_2 .

With the above information ΔF° and ΔH° are calculated to be

$$\Delta H^\circ_{25^\circ\text{C}} = 3465.7 \text{ kcal/gmole } \text{C}_{30}\text{H}_{22}\text{O}_4$$

and

$$\Delta F^\circ_{25^\circ\text{C}} = 3510.2 \text{ kcal/gmole } \text{C}_{30}\text{H}_{22}\text{O}_4$$

The equilibrium constant at this temperature may be calculated from Equation 1.

$$K_{25^\circ\text{C}} = e^{\frac{(3510.2 \text{ kcal})}{R \cdot T}} = e^{5890} = 10^{2555}$$

If the usual assumptions for the pressures of this reaction (less than 1 atmosphere) are made, the activity coefficients equal one so we can write:

$$K_{25^\circ\text{C}} = P_T^{6.5} \frac{X_{\text{CO}_2}^{30} \cdot X_{\text{H}_2\text{O}}^{11}}{X_{\text{O}_2}^{33.5} \cdot X_{\text{C}_{30}\text{H}_{22}\text{O}_4}} \quad (6)$$

where P_T is the total pressure of the reaction and $P_i = X_i P_T$ where

P_i = partial pressure of component i at equilibrium and X_i = mole fraction of component i after reaction.

Because of the size of K , we may conclude that the reaction goes to completion. However, for clarity, let us calculate the equilibrium concentration.

$$\begin{aligned} \text{Let } A_o - z &= \text{moles of } C_{30}H_{22}O_4 \text{ at equilibrium} \\ B_o - 33.5z &= \text{moles of } O_2 \text{ at equilibrium} \\ 11z &= \text{moles of } H_2O \text{ at equilibrium} \\ 30z &= \text{moles of } CO_2 \text{ at equilibrium} \end{aligned}$$

then

$$K = \frac{P_T^{6.5}}{(A_o + B_o + 6.5z)^{6.5}} \cdot \frac{(11)^{11} \cdot (30)^{30}}{(A_o - z) \cdot (B_o - 33.5z)^{33.5}} \quad (7)$$

Let $A_o = 1$, $B_o = 33.5$, and $P_T = 1$ atmosphere (Stoichiometric conditions at one atmosphere).

Calculation of z yields

$$10^{2555} = \frac{10^{-6}}{(1 - z)^{34.5}}$$

$$1 - z \approx 10^{-74}$$

$$z = 1 - 10^{-74} \approx 1$$

which means that the reaction is complete at equilibrium at one atmosphere. Because of the form of Equation 7, it is seen that as the pressure of reaction is decreased, the reaction tends more toward completion since $\frac{K}{P_T^{6.5}}$ increases.

If the reaction temperature is increased from 25°C to 325°C (which corresponds to 585°F), the equilibrium composition can be estimated by assuming $\frac{\Delta H}{T_o} = 0$ (which is a good assumption since s is small) and by estimating the change in ΔF° by the change in ΔH .

$\Delta H^\circ_{325^\circ\text{C}} - \Delta H^\circ_{25^\circ\text{C}} = \Delta C_p \Delta T$ where ΔC_p is the mean specific heat of all the components in the reaction.

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$$\Delta C_p = 11 \times C_p H_2O + 30 \times C_p CO_2 - 33.5 C_p PO_2 - C_p C_{30}H_{22}O_4$$

$$= 11 \times 8.2 + 30 \times 10.1 - 7.2 \times 33.5 - C_p C_{30}H_{22}O_4$$

$$= (152 - C_p C_{30}H_{22}O_4) \text{ cal/gmole } ^\circ F$$

By letting $C_p C_{30}H_{22}O_4 = 0$, we will find the maximum change in ΔH .
Therefore:

$$\Delta(\Delta F) = .152(300) \text{ kcal/gmole}$$

$$\Delta F_{325^\circ C} = (-3510.2 + 45.6) \text{ kcal/gmole}$$

$$\Delta F_{325^\circ C} = -3464.6 \text{ kcal/gmole}$$

$$K_{325^\circ C} = e^{\frac{3464.6 \text{ kcal}}{R \cdot T}}$$

$$K_{325^\circ C} = 10^{1260}$$

In this case

$$(1-z)^{34.5} = 10^{-1266}$$

$$1-z = 10^{-36.7}$$

or

$$z = 1$$

Again in this case, a lower pressure favors the reaction.

The conclusion which can be drawn from the above calculations is that equilibrium considerations indicate that a very large potential exists for the reaction of Convalex 10 in the temperature and pressure ranges of interest.

Although we were able to estimate the change in standard state free energy for Convalex 10 by the method of Franklin, we could not do this for the silicon base oils such as DC 705 since he has not considered these groups.

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With a considerable amount of additional work, ΔF^0 of these reactions could be estimated. It is not necessary to do this because of the similarities between these oils, one can predict that $-\Delta F^0$ will be very large for reactions involving these oils and the conclusion that a large potential will exist for the other reactions will also apply.

In the experimental work involved in this project, none of these oils were prone to explosion as the above analysis indicates they should be. The reason why they are not will be brought out in the following section in the description of reaction rates. Equilibrium considerations are comparable to finding the potential energy difference between the top of a mountain and sea level. The kinetics of reactions or reaction rate considerations are comparable to finding a path down that mountain.

B. Theories of Combustion⁽⁴⁾

Combustion is a chemical reaction under conditions of progressive self-acceleration, due to the accumulation in the system of heat or of catalyzing active intermediate products. In the first case, the combustion is termed thermal, in the second, diffusional or chain combustion.

Either type of combustion is controlled by the reaction rate. The reaction rate is a function of the reaction mechanism. Each of these types of combustion will be described briefly in this section to indicate the role of the reaction rate in determining explosion limits.

1. Thermal Theory of Combustion

The equation of heat transfer in a gas for small values of the Grashof criterion, which means that transfer is only conductive, is given by:

$$C_p \rho \frac{\partial T}{\partial t} = \text{div } \lambda \text{ grad } T + q^1 \quad (8)$$

where C_p is the specific heat

ρ is the density

T is the temperature

t is the variable, time

λ is the coefficient of heat transfer
and q is the heat density of the sources.

In the case of a reaction, the heat source present is the heat released by the reaction. This term, q is a function of the composition, pressure, and temperature of the gas.

Equation 8 clearly illustrates that the concept of an ignition temperature is not a concept of a unique quantity but rather one that depends upon heat transfer. If the temperature in a vessel initially at T_0 increases a small amount and then becomes constant, this is not considered an explosion but rather a reaction. If, however, the temperature increases without limit, the reaction is termed an explosion. The temperature at which the transition between these two regions occur is termed the ignition temperature. Since the term q is a function of pressure and gas composition, so is the ignition temperature.

The form usually assumed for q is

$$q = Q z \text{ EXP } (-E/RT) \quad (9)$$

where Q and E are constants determined from experiment, z is a function of pressure and composition, and R is the universal gas constant. This is referred to as the Arrhenius Law and does not necessarily represent the heat released in the gas volume because $z \text{ EXP } (-E/RT)$ does not necessarily represent the reaction rate of a reaction. For purposes of illustration we will assume this is representative.

Substitution of Equation 9 into Equation 8 yields:

$$C \int \frac{\partial T}{\partial t} = \text{div} \lambda \text{ grad } T + Q z \text{ EXP } (-E/RT) \quad (10)$$

Since Equation 10 is very difficult to solve mathematically, two different methods of approximation have found acceptance. The first method, called the stationary method, assumes that $(\partial T / \partial t) = 0$ and that the boundary condition at the wall is $T = T_0$. With these assumptions and assuming λ is independent

of temperature, Equation 10 reduces to

$$\nabla^2 T = -\frac{Q}{K} z \text{ EXP}(-E/RT) \quad (11)$$

where ∇^2 is the La Placian operator. The solution of this equation gives the stationary temperature distribution in a reacting mixture.

The concept of an explosion with the above assumptions is tantamount to the impossibility of the solution for a temperature distribution. It can be seen at this point that the reaction rate will determine this condition even though the calculations of the previous section indicate that a reaction should take place.

The second method, called the non-stationary theory, assumes that the temperature is constant throughout the container and the heat transfer away from the vessel is

$$q_{\text{conv}} = \alpha_s (T - T_o) \quad (12)$$

where α the convective heat transfer coefficient, s is the surface area and T_o is the temperature of surroundings. In this case, Equation 10 becomes

$$w C_p \frac{dT}{dt} = W Q z \text{ EXP}(-E/RT) - \alpha_s (T - T_o) \quad (13)$$

where w is the volume of the container. The criterion of explosion in this case is equivalent to finding a solution which states that the temperature becomes infinite as time increases. This theory is usually illustrated by the explanation of Fig. 1. ⁽⁵⁾

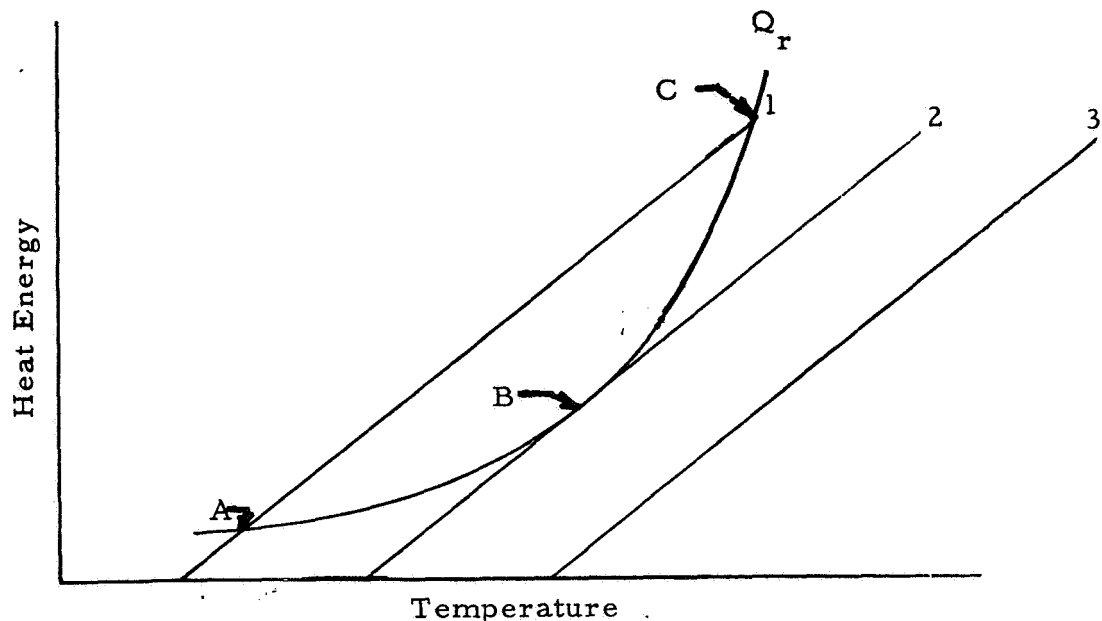


Fig. 1 THERMAL THEORY OF EXPLOSION-NON
STATIONARY

Curves 1, 2, and 3 represent three possibilities of the convective heat transfer away from the vessel and Q_r represents the heat released from the reaction. If the heat transfer from the vessel is represented by curve 1, then it is seen that any initial temperature below T_A would raise the temperature to T_A . Any initial temperature between T_A and T_C would lower the temperature to T_A . Any temperature above T_C would cause an explosion since the temperature would increase without limit. If the heat transfer from the vessel were represented by curve 2, any temperature below T_B would tend toward T_B . Any temperature above T_B would cause an explosion. If curve 3 represented the heat loss to the surroundings, any temperature would cause an explosion.

This analysis brings out the fact that an ignition temperature is not only dependent upon concentration and pressure but upon heat transfer away from the vessel. This concept plays a very important role in the theory of spark ignition since a large amount of heat is added to a small volume of gas.

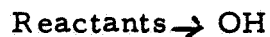
2. Theory of Chain Reactions

It was seen in the previous section that thermal explosions depend

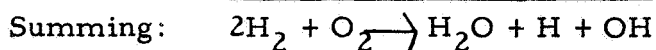
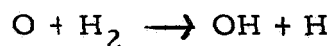
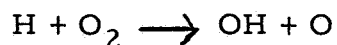
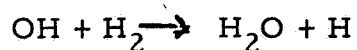
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upon the heat release of the reaction for propagation and upon the temperature rise for acceleration of the reaction. Chain reactions do not depend upon a temperature rise for acceleration of the reaction. An example of a chain reaction for the hydrogen-oxygen reaction is shown in the following equations:

Initiating Step



Chain Steps



which indicates we have a chain branching step. Once one OH is formed, it starts the chain steps which are self sustaining. It is seen that the progression of the reaction is controlled by the chain branching steps. This reaction mechanism is by no means representative of chain reactions in general since three or more chain producing steps may be present in a reaction.

The hydrogen-oxygen reaction exhibits both thermal and chain branching mechanisms. The explosion limits of this reaction are shown in Fig. 2⁽⁶⁾

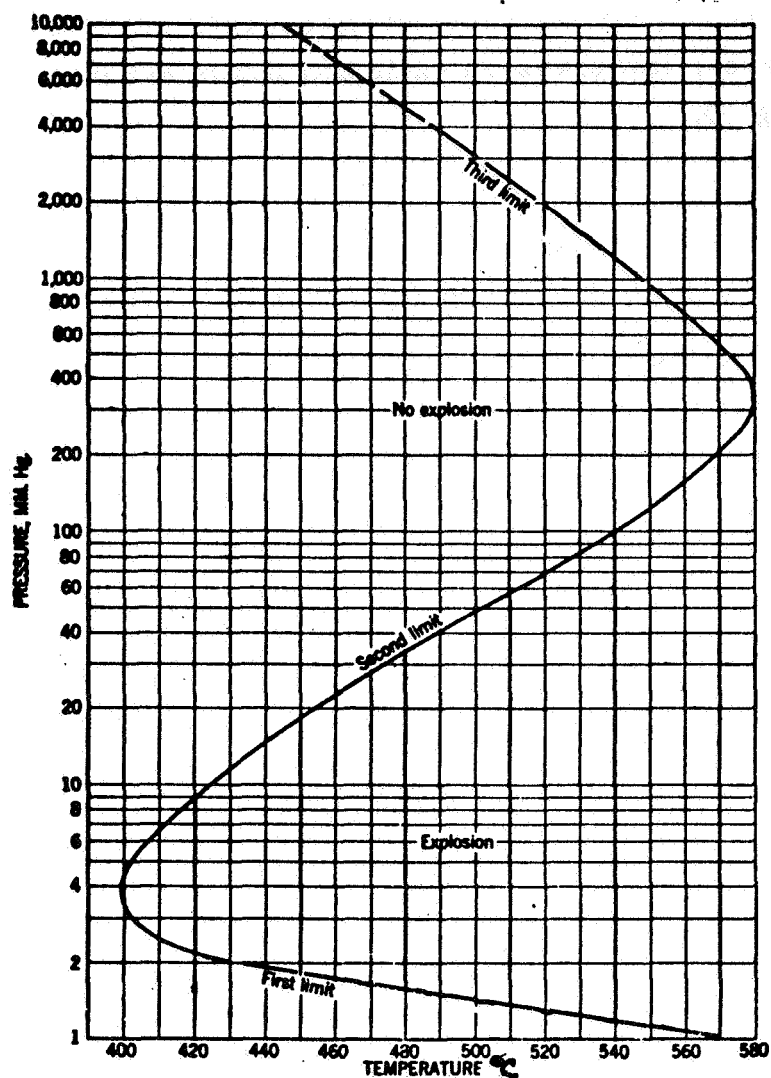


Fig. 2 EXPLOSION LIMITS OF A STOICHIOMETRIC HYDROGEN-
OXYGEN MIXTURE IN A SPHERICAL
KCL-COATED VESSEL OF 7.4 CM.
DIAMETER

It should be noted that the explosion limit curve for this reaction is not representative of reactions in general, each exhibits a different form. However, in a multitude of cases, low pressure explosion limits exhibit a shape similar to that shown in Fig. 3.

3. Detonation

In addition to the thermal and chain reaction explosions, detonation may be observed for gases which normally exhibit only a flame progression.

It is known that the transition from slow burning to detonation in gaseous fuel mixtures occurs during the process of accelerated flame propagation. According to existing theories, in normal propagation of the flame from the closed end of a tube, the expansion of the combustion products causes motion in the fresh mixture ahead of the flame front. This leads to turbulence of the mixture, an increase in the flame surface due to nonuniformity of the distribution of the flow velocity over the cross section, and acceleration of the flame. Accelerating progressively, the burning creates an adiabatic compression wave. The latter, as the steepness of the flame front increases, produces a shock wave and then a detonation wave. ⁽⁷⁾

C. Practical Application of the Theories of Combustion

It is seen that the existence of explosions depends entirely upon heat transfer and reaction rates, once a reaction has been shown to be possible from equilibrium considerations. Therefore, in order that results of combustion experiments be applicable in general, the reaction rates should be obtained. Reaction rates, presently, cannot be calculated satisfactorily and, therefore, must be measured experimentally. Standard techniques are usually restricted to a narrow range of operation. To obtain the most amount of explosion information in a given time, combustion cells are frequently used to obtain explosion limits directly.

D. Explosion Limits with an External Ignition Source

It has been postulated that in order for an explosion to occur because of energy dissipation in a spark, a flame volume of critical size must be established. ⁽⁸⁾

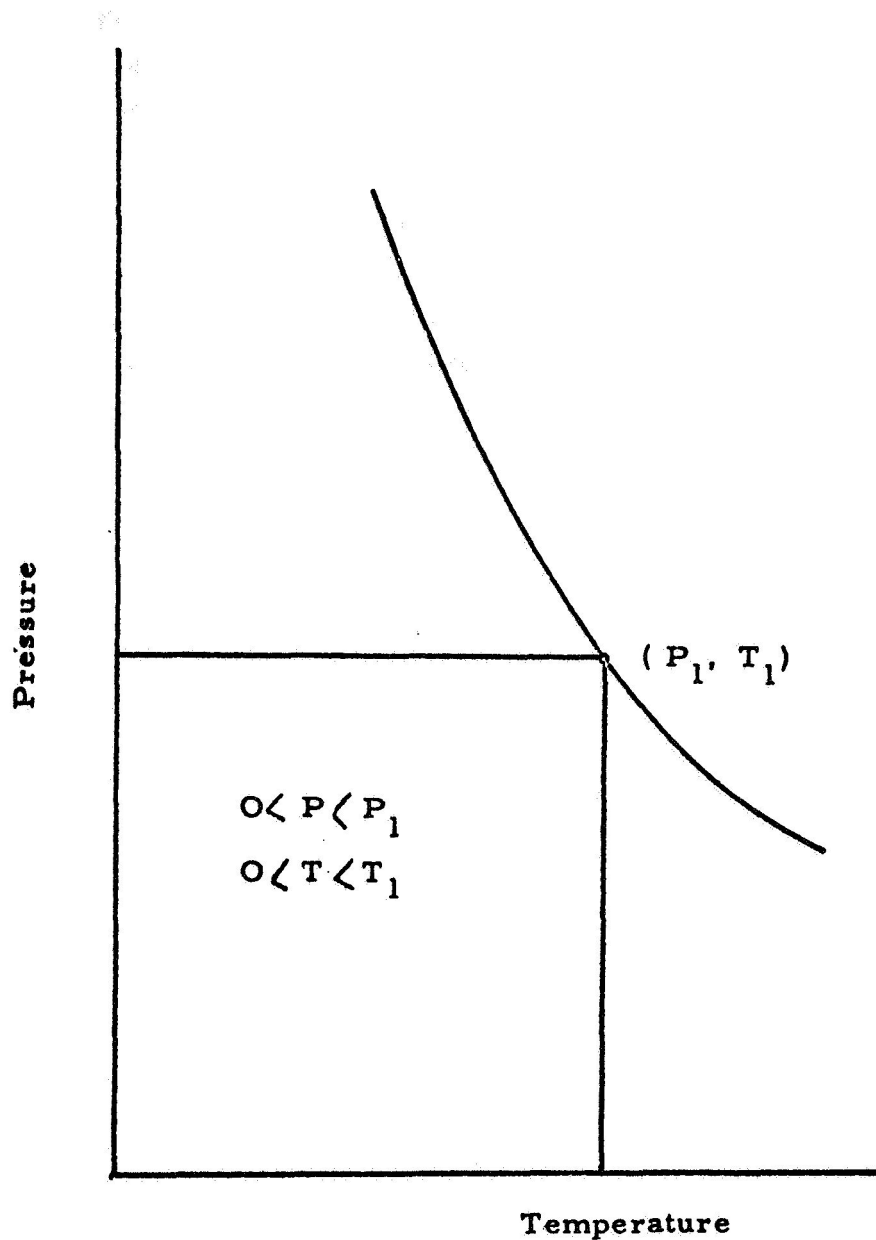


Fig. 3 TYPICAL LOW PRESSURE EXPLOSION LIMIT CURVE

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A minimum ignition energy is required to establish a flame of critical size. If the energy is insufficient and hence the minimum size is not attained, the ambient unburned gas serves as a quenching agent by dissipating the heat of the reaction zone. As mentioned previously, this can be seen from Equation 13 and Fig.1 since the thermal theory of explosion applies to this case.

Whether the flame develops to the steady state depends on the size to which the inflamed volume has grown at the time when the temperature at the origin has decreased to the order of the normal flame temperature. It is assumed that the combustion wave develops in spherical symmetry. The energy within the spark volume decreases very rapidly due to the flow of heat to the ambient unburned gases.

It has been shown⁽⁸⁾ that immediately after a spark discharge, virtually all the spark energy resides in the gas and that the rate of subsequent cooling of the gas by the electrode material is negligible in the time required for the formation of the flame of a critical size, except in the case where the electrodes are spaced too closely.

An estimation of the temperature attained in a spark may be calculated by assuming that the energy is dissipated in the mass initially in the spherical volume between the electrodes to obtain a representative value of temperature increase. For this experiment the following conditions were chosen: a pressure of 100 mm, an initial temperature of 700°F, a specific heat of 8.2 cal/gmole°C for oxygen, an electrode spacing of 0.1 in, and an energy dissipation of 6 millijoules. The temperature increase was calculated to be 1835°F. This temperature increase is probably a low value since the volume dissipation is probably smaller than that assumed. However, this value is assumed to be representative.

E. Theory of Combustion in the Presence of Catalysts

As mentioned previously, combustion of a gas depends upon the heat transfer and reaction rates. The presence of a catalyst does not change this dependence, however, it may change the reaction rate by changing the reaction mechanism; thus, promoting or suppressing a reaction. Since a very large reaction potential exists for the oils investigated in this program,

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it may be expected that the presence of the right catalyst could change the reaction mechanism and promote explosions. Because of this fact, the effect of catalysts should be evaluated very carefully and metals incorporated in the Space Environmental Chamber should be restricted to those metals which have been evaluated.

V COMBUSTION EXPERIMENTS

The purpose of the combustion phase of this project was the study of five diffusion pump oils and their relative hazard potentials when used in a manned environmental space simulation facility. These oils were subjected to rigorous testing under the various conditions necessary to determine their hazard potential. Their explosion limits were determined by subjecting them to a wide range of conditions calculated to favor explosions.

DC-705 and Convalex 10 both showed enough resistance to these tests to warrant further investigation into their ability to resist any catalytic initiation of an explosion. DC-705 was selected as the most desirable diffusion pump oil and was further subjected to a very wide range of experimental conditions. In addition, sparks were used in attempts to ignite mixtures of DC-705 and oxygen.

Several physical properties of each oil were considered of special interest to the performance of this work. These properties are included in Appendix C. Among these are vapor pressure curves, molecular formulas, stoichiometric equations etc.

A Results

1. DC-705 Data Obtained with the Glass System

The results of combustion testing performed with DC-705 conducted in accordance with the procedure described for the new glass system in Appendix B are tabulated in Table 1 of Appendix A. The results indicate that no spontaneous explosion occurred under the conditions investigated. The range of conditions investigated are summarized in Table 4.

Explosions were observed in the glass system when a spark was dissipated under certain conditions. The results of the spark tests are tabulated in Table 1 in Appendix A. The conditions under which explosions occurred are listed also in Table 5 and plotted in Fig. 4.

TABLE 4
EXPERIMENTAL CONDITIONS INVESTIGATED
FOR DC-705

Reservoir Temperature (° F)	Combustion Cell Temperature (° F)
288	320
	440
	540
	800
390	422
	450
	500
	550
	620
	700
	800
	900
440	460
	530
	620
	740
	750
	800
	850
570	630
	715
	800
	850
	900

In the last test, the cell was initially filled to 256 torr oxygen at 752° F and the temperature of the cell was raised until the vapor pressure exceeded 100 torr.

TABLE 5

SUMMARY OF COMBUSTION DATA
FROM SPARK IGNITION EXPERI-
MENTS. DC-705 DIFFUSION PUMP
FLUID

Experi- ment No.	Bomb Tempera- ture (° F)	Total Pressure (m m)	Spark Energy (Milli- joules)	Concentration Factor* <i>Ø</i>
11 (a)	630	73.5	6.76	9.57
11 (b)	715	73.5	6.76	24.7
9 (a)	740	70.2	5.2	1.18
9 (b)	745	25.6	6.76	1.51
9 (c)	735	24.0	6.76	2.84
9 (d)	735	20.5	10.2	1.99
9 (e)	800	29.9	3.96	1.06
11 (c)	800	48.8	6.76	36.3
10 (b)	850	37.6	6.76	7.85
10 (c)	850	25.6	6.76	11.5
11 (e)	890	35.0	6.76	8.45

* This factor is defined at the beginning of Appendix A.

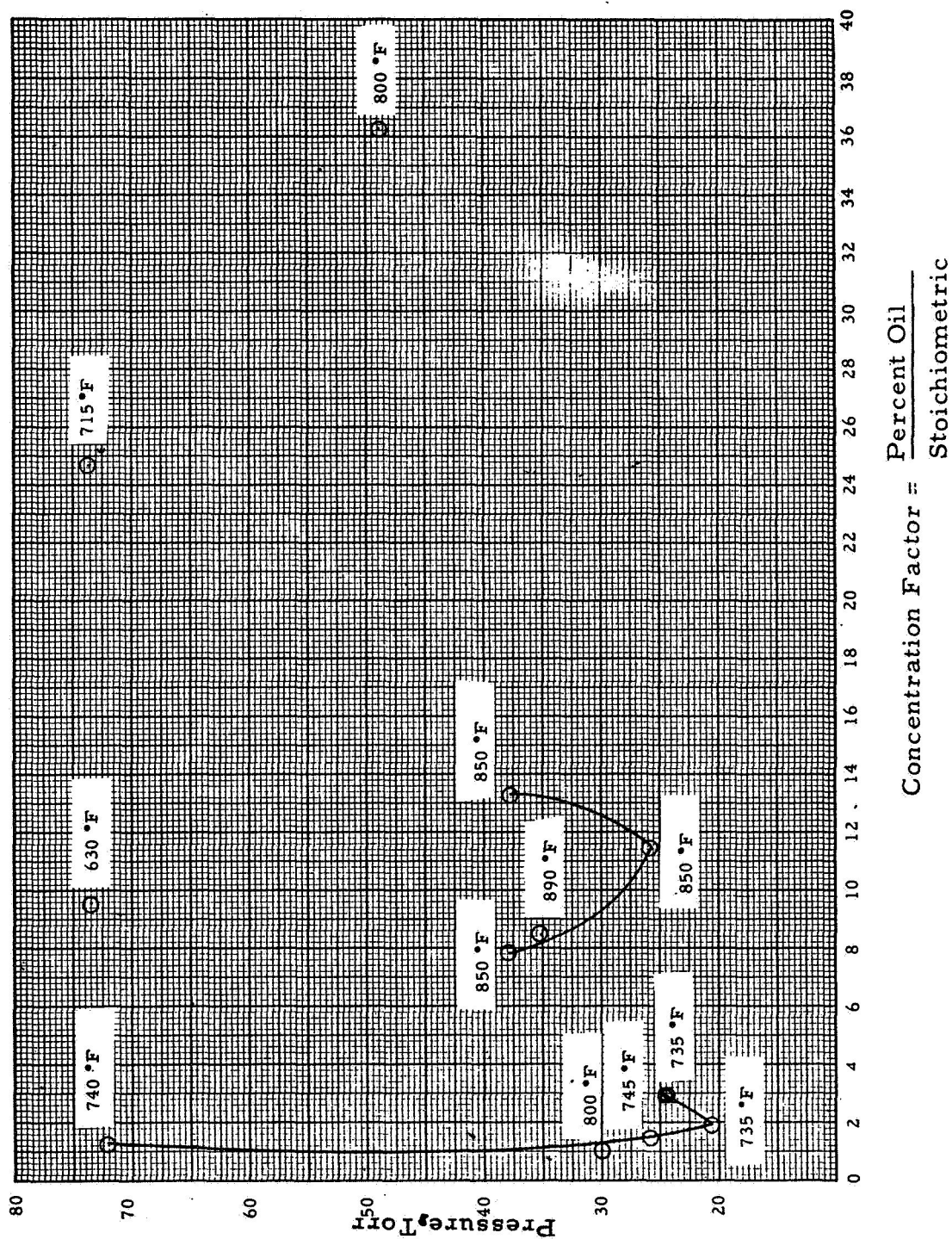


Fig. 4 EXPLOSION PRESSURES AS A FUNCTION OF CONCENTRATION FACTOR

2. Stainless Steel System

The combustion tests, conducted in the stainless steel combustion cell according to the procedure outlined in Appendix B, did not indicate any explosion for DC-704, DC-705, Convalex 10, or TCP under the conditions investigated. Explosions were observed for DC-704 and Convoil 20. A summary of the results of these tests is shown in Table 6.

TABLE 6

SUMMARY OF THE RESULTS OF THE COM-
BUSTION TESTS IN THE STAINLESS STEEL
COMBUSTION CELL

<u>Range of Conditions Investigated</u>			<u>Observed Explosions</u>		
Oil	Pressure Range (torr)	Tempera- ture (°F)	Total Pressure (torr)	Cell Tempera- ture (°F)	Reservoir Tempera- ture (°F)
DC-705	780	580-900	No Explosions		
Convalex 10	760	700-900	No Explosions		
DC-704	760	600-900	Explosions at		
			708	621	506
			455	806	513
			633	805	512
			300	893	516
			40	897	513
			No other Explosions		

TABLE 6 (Con't)

<u>Range of Conditions Investigated</u>			<u>Observed Explosions</u>		
Oil	Pressure Range (torr)	Tempera- ture Range (° F)	Total Pressure (torr)	Cell Tempera- ture (° F)	Reservoir Temperature (° F)
TCP	400	600 900	No Explosions		
Convoil 20	400	600-900	Explosions at		
			250	707	534
			428	708	535
			530	709	535
			403	708	542
			607	802	512
			148	796	512
			No other Explosion		

The specific conditions investigated are tabulated in Tables 2 to 6 inclusive in Appendix A.

3. Initial Glass Experiments

Combustion experiments, conducted in the initial glass system according to the procedure outlined in Appendix B, indicated explosions of Convalex 10, DC-704, DC-705 and the results are shown in Table 7. After consideration of the experimental procedure, these data appear to be incorrect and should be disregarded since these explosions were never reproduced in subsequent work in the newer systems. At present, it has been concluded, that since no cold trap was used from the mechanical pump, the explosions were due to backstreaming of mechanical pump oil.

TABLE 7

PRELIMINARY EXPLOSION DATA NOW
CONSIDERED INCORRECT

Oil	Pressure (torr)	Temperature (°F)
Convalex 10	190	800
Convalex 10	444	745
DC-704	550	680
DC-705	632	745

4. Catalytic Experiments

The results of the catalytic experiments, performed according to the procedure described in Appendix B, did not positively indicate that any explosion occurred during a fifteen day experiment. After fifteen days sparks of an energy of 8.35 millijoules were dissipated in each of the combustion cells and these produced no explosions. Some of the catalytic combustion cells failed because of a physical malfunction. The results of the catalytic tests are described in Table 8.

5. Detonation Experiments

Each of the five oils were subjected to a detonation experiment with pressed tetryl pellets used as boosters. The results showed that these oils would not detonate.

B. Discussion

1. Selection of the Optimum Diffusion Pump Oil

The main objectives of this project was to evaluate the hazard potential of pre-selected diffusion pump oils under certain conditions which approximate and/or magnify greatly conditions which could develop in the

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TABLE 8

SUMMARY OF RESULTS OF THE CATALYTIC COMBUSTION EXPERIMENTS

Experiment	Representative Temperature (°F)	Initial Pressure on 6/1/63 (torr)	Final Pressure on 6/16/63 (torr)	Pressure Increase (torr)	Remarks
No catalyst DC-705	377	414	419	+5	No explosions were indicated and no leaks were observed. The DC-705 was very clean at the end of the test.
Stainless Steel in DC-705	431	415	305	-110	A pressure decrease was observed thus indicating no explosion occurred. The liquid was slightly yellow in color.
Steel in DC-705	450	384	368	-16	A slight pressure decrease was observed indicating no explosion occurred. The liquid was slightly yellow.
Copper in DC-705	470	305	226	-79	A slight pressure decrease was observed indicating no explosion. During the initial starting, the oil melted some rubber tubing and contaminated the sample and closed up the entry to the cell. It is doubtful that any oxidant but air was in this cell.

Experiment	Representative Temperature (°F)	Initial Pressure on 6/1/63 (torr)	Final Pressure on 6/16/63 (torr)	Pressure Increase (torr)	Remarks
Copper in DC-705 (Cont. from previous page)					(con't) Inadvertently, this heater was left on after the test was completed. Some time between 6/16/63 and 6/20/63 an explosion occurred in this cell.
Aluminum in DC-705	386	400	593	193	It is assumed that this pressure rise was gradual and thus was due to leaks into the system rather than a reaction. The pressure gage did not record properly during this period so that one cannot be absolutely certain that a small explosion did not occur.
No catalyst Convalex 10	440	765	760	-5	This cell did not seal properly but this test was considered expendable. Convalex 10 turned black after a few days of operation.
Stainless Steel in Convalex 10	This combustion cell did not function properly. However, no reactions occurred in the stainless steel combustion cell tests so it is concluded, stainless steel will not cause a reaction.				
Steel in Convalex 10	448	381	361	-20	Pressure decrease indicates that an explosion did not occur. The Convalex 10 turned black during the first few days of operation.

TABLE 8 (con't)

Experiment	Representative Temperature (°F)	Initial Pressure on 6/1/63 (torr)	Final Pressure on 6/16/63 (torr)	Pressure Increase (torr)	Remarks
Copper in Convalex 10	341	417	493	+ 76	It is assumed that this pressure rise is due to small leaks into the system. The pressure gage did not record properly and the above statement is based on daily observation.
Aluminum in Convalex 10	418	415	648	233	The pressure gage did not record properly. This pressure rise can be due to a reaction or to leaks. The pressure was observed visually each day and this seemed to be a slow pressure rise. However, the results of this test must be considered inconclusive

Manned Space Environmental Chamber. If a hazard exists, then a method of circumventing this problem should be found.

All five diffusion pump oils were examined for explosions under various conditions of temperature, pressure, and concentration. Knowledge of these and other results indicates that DC-705 and Convalex 10 are the best of the five oils for use in a diffusion pump. Because of the prohibitive cost of Convalex 10, DC-705 seems to be the best oil to use. The reason for making these statements should be apparent from the following discussion:

The low pressure explosion limit of many fuels is an inverse relation of pressure and temperature at constant composition is shown in Fig. 3. It is reasonable to assume that the combustion properties of these oils are similar. Therefore, if an explosion does not occur at pressure P_1 , and temperature T_1 , the logical conclusion to draw is that an explosion will not occur at a condition such that $0 < P < P_1$ and $0 < T < T_1$ at a constant composition.

Convoil 20

Convoil 20 is a hydrocarbon and for this reason, its oxygen compatibility is uncertain. In fact, explosions did occur with Convoil 20 and not with the other oils. Infrared analysis at ARF showed that only hydrocarbons are present in the oil, and gas chromatography showed that these consisted of a petroleum cut between C_{20} and C_{30} . Analysis showed that a large amount of thermal decomposition had occurred in the oil, breaking it down into more volatile constituents. Explosions did not occur at 600°F but did occur at 700°F. From the previous discussion, we may conclude that it is safe to use Convoil 20 for the conditions.

$$0 < P < 400 \text{ torr}$$

$$0 < T < 600^\circ\text{F}$$

no matter what the stoichiometric ratio is: Other considerations affect this conclusion since the liquid oil at a temperature of 533°F showed evidence of a large amount of decomposition. This temperature approaches the operating temperature in a diffusion pump and, therefore, limits the use of this oil as a diffusion pump fluid. Consider, also, that a fire caused by degradation of other materials in the simulated-space chamber would be propagated with this oil because it decomposes. Therefore, in our opinion, Convoil 20 does not meet the requirements of NASA.

Tricresyl Phosphate

The vapor pressure of TCP was measured by an outside organization with use of an ebulliometer. The results indicate that thermal decomposition begins at 433° F. Although no explosions were obtained with this oil, thermal decomposition at this temperature impedes the use of this oil, and, therefore, it is not recommended.

DC-704

Evidence of explosion of DC-704 was observed in the stainless steel apparatus. As indicated in Table 3, Dow Corning advertised DC-705 as a better over-all pump fluid than DC-704, and we concur with this opinion.

DC-705

No evidence of explosion of DC-705 was observed in the stainless steel apparatus. The delivery time of DC-705 is about two weeks and the cost is about \$200/gal in 5-gallon lots. The vapor pressure is below that of DC-704 so that this oil is considered a very good choice of oils with a minimum hazard potential.

Convalex 10

No evidence of explosion of Convalex 10 was observed in the stainless steel apparatus. The delivery time and cost of this oil are prohibitive so that this oil is not recommended. It was reported by the Chemistry Division of Armour Research Foundation, that Convalex 10 does not decompose more than two percent even when heated to 1000° F. However, at about 450° F this oil begins to turn black and may be considered objectionable for this reason.

With the aid of the above considerations, the oils used in the vacuum phase were selected. Convalex 10, DC-704, and DC-705 were selected for the 6 inch pump and DC-704 and DC-705 were selected for the 35 inch diffusion pump.

DC-705 was selected as the best oil from the stainless steel cell experiments, but before we could recommend this oil without reservation we had to subject it to a thorough investigation. The glass system was used for this purpose. Approximately five times as much data was taken on DC-705 than on the other oils. Pressure, temperature, and concentration effect upon ignition tendencies were investigated over wide ranges as evidenced in Table 1 of Appendix A. Energies derived from spark gaps were dissipated throughout the range of investigation. No spontaneous ignitions resulted under any conditions investigated thus reinforcing our opinion of DC-705.

Spark dissipations resulted in explosions as evidenced in Table 2 and Fig. 4. All but one of these explosions occurred above 700°F. It is reasonable to assume that DC-705 might explode above 700°F because the thermal decomposition begins at this temperature.

We had an outside organization measure the vapor pressure of DC-705 by use of an ebulliometer. It confirmed the vapor pressure data reported by Dow Corning. In addition to learning this fact, the thermal decomposition temperature was reported to us. Thermal decomposition is important because a large molecule like DC-705 must decompose before it reacts.

It is our considered opinion from the examination of the data obtained, that a mixture of DC-705 and oxygen will not explode without some outside source of ignition, if the total pressure is below 450 torr and 600°F.

2. Catalytic Investigation

The objective of the catalytic experiments was to demonstrate the effect of four common construction metals on the safety limits of the two least hazardous diffusion pump oils.

The experiments were designed to aid any catalytic effects as much as possible within the limits of the worst conditions that could reasonably be anticipated in the system described by NASA.

The nuclei of the catalytic combustion system were ten combustion cells, as shown in Fig. 5 of Appendix B.

Since the pressure and temperature chosen were subcritical for explosive reactions, any large scale reaction, whether slow or fast, would be indicative of catalysis in the cell.

A qualitative comparison of the various results described in Table 5 indicates some interesting possible conclusions.

Metal shavings were used in the cells to present maximum surface area to the vapor-oxygen mixture. This increased potential for catalytic effects of stainless steel on DC-705 or Convalex 10 over any possible effects generated by the stainless steel combustion bomb seemed to have very little effect. It may be said, therefore, that the possibility of a hazard existing because of stainless steel in a diffusion pump system is minimal for these two oils.

The cell containing copper and DC-705 was inadvertently contaminated with melted vacuum hose. A few days after the catalytic experiments had been terminated, the laboratory was checked. The heater on this cell had not been turned off and it appeared that a large explosion had occurred in the interim. It is believed by the observer that the reaction was due more to the contamination than to the copper. If a reaction did in turn occur, it would indicate that contamination of the diffusion pump oil would constitute a very definite hazard potential.

The pressure monitoring devices failed in large. Only two out of ten worked properly. When the cells were initially installed, they were

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checked out thoroughly and seemed to work satisfactorily. However, some-time after the experiment was begun, the resistance element, which was to measure the height of the mercury by means of the shorting effect of the mercury presumably became coated with oil. This rendered the pressure monitor useless except as a visual read out device. Measurements were made visually at regular intervals.

Since no large pressure increase occurred in any one day, the catalytic effect of these metals is probably minor with respect to DC-705 and convalex 10.

3. Detonation Experiments

As described in the theory section, some explosions are started or propagated by a pressure impulse. Such an explosion may be called a detonation. It is not necessary for the reactant to combine with oxygen for a detonation to occur. It is possible for the reactant to decompose exothermally and create a phenomenon similar to an explosion. We had been informed of an accident which was caused by detonation of a substance with a molecular structure similar to Convalex 10. For this reason, we decided that the five oils should be tested for detonation.

The test was conducted by the use of ordinary detonation techniques. The pressed tetryl pellets used as boosters are the standard types used in the kind of testing of all secondary explosives. Our test results showed that no detonations occurred with any of these oils. We conclude that conditions which are less severe than those investigated cannot result in the condensed phase detonations of the oils being considered here. Conditions in a diffusion pump fall in this category so that detonation is discarded as a possible hazard.

VI. HIGH VACUUM PHASE

A. General Remarks on Backstreaming

Backstreaming of vapors from diffusion pumps is a highly controversial and poorly understood phenomenon. The discussion here is limited to the evolution of pump fluid into a vacuum chamber from an oil diffusion pump. Many researchers have studied the problem and many techniques have been employed to quantitatively define backstreaming rates. The published results are contradictory, and no standards are available for comparative evaluation. The backstreaming rates listed in Table 9 illustrate the experimental results cited in the literature. The accumulation rates and equivalent pressures have been determined by these writers from the following which may be found in any text on kinetic gas theory:

Accumulation rate, A;

$$A = \frac{G \times 6.023 \times 10^{23}}{d \times M} \left(\frac{\text{Monolayers}}{\text{Minute}} \right)$$

Equivalent Pressure, P;

$$P = 17.14 G \sqrt{\frac{T}{M}} \text{ (torr)}$$

where

G = backstreaming rate (g/cm² min)

d = area covered by one molecule, 2×10^{-13} (cm²) (Estimated value)

M = molecular weight of vapor (g/mole)

T = temperature of vapor (°K)

The technique generally employed for determining backstreaming rates is to place a condensation plate over the pump throat and to analyze the collected fluid volumetrically or gravimetrically. Improvements in pumps, baffles and pump fluids have reduced backstreaming to the point where this technique is inapplicable. Additional complexity stems from recent findings¹³ which indicate that the backstreaming rate during start-up and shut down may be many orders of magnitude greater than the steady rate, thus making steady state rate measurement difficult.

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TABLE 9
PUBLISHED BACKSTREAMING RATES

Pump Dia. (in.)	Auxilliary Equipment	Fluid	Backstreaming Rate	Accumulation Rate ($\frac{\text{Monolayers}}{\text{Min.}}$)	Equivalent Pressure (torr)	Ref.
6	None	(a)	$0.03 \text{ mg/cm}^2 \text{ mn}$	2040	6.8×10^{-5}	12
6	Cold Cap	(a)	$5 \times 10^{-4} \text{ mg/cm}^2 \text{ mn}$	31	1.1×10^{-8}	12
6	5°C Baffle	DC 704	$6 \times 10^{-7} \text{ mg/cm}^2 \text{ mn}$	0.037	1.4×10^{-12}	13
6	5°C Baffle	DC 705	$5 \times 10^{-7} \text{ mg/cm}^2 \text{ mn}$	0.031	1.1×10^{-12}	13
6	5°C Baffle	OS 124	$5 \times 10^{-8} \text{ mg/cm}^2 \text{ mn}$	0.0031	1.1×10^{-13}	13
6	None	Conva- lex 10	0.0126 g/hr.	52.5	1.9×10^{-7}	14

(a) Unspecified

mg = milligram

g = gram

cm = centimeter

mn = monolayer

hr = hour

1. Backstreaming tests with the PMC 50,000 Pump

In light of the above discussion, it was determined that backstreaming rates for the equipment used on this program could not be made by existing techniques within time and funding allocations. Various techniques which could be employed were investigated and are listed below:

- a. Wettability and contact angle measurements
- b. Ellipsometry
- c. Infra red spectroscopy
- d. Electrical resistivity
- e. Gas chromatography
- f. Mass spectroscopy
- g. Radiometric measurements

The first two techniques were selected as being the most promising and were used in tests with the PMC 50,000 diffusion pump. Both techniques can be used to measure film thicknesses of molecular dimensions, including partial coverage. The techniques are described briefly below.

a. Wettability and contact angle measurement

The degree of wettability of a surface by a liquid is a function of the relative surface energies involved and can be described by a single factor, the critical surface energy of the system. A surface with a given surface energy can be wet only by a liquid with a lower surface tension. The critical surface energy at a liquid-solid interface may be measured by observing the contact angles formed when drops of liquids of varying surface tension are placed on the surface. A plot of the cosines of the angles formed versus the surface tensions of the liquids will determine a straight line whose intercept with the line $\text{Cosine} = 1$ is the critical surface energy of the system. Since clean metal surfaces have surface energies in the hundreds of dynes per cm, and contaminated surfaces exhibit much lower surface energies, it is possible to determine the degree of contamination by suitable contact angle measurements.

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b. Ellipsometry

When linearly polarized light is reflected obliquely from a specular metallic surface, the components of the reflected beam parallel and perpendicular to the plane of incidence are unequal both in amplitude and in phase, and therefore the reflected beam is elliptically polarized. The relative amplitude and phase are conveniently represented as a complex reflection. These parameters are remarkably sensitive to the presence of thin dielectric films on the mirror. Average thickness changes of as little as 0.1\AA have been detected in thick layers under ideal conditions. In the measurements made for this program, precision was approximately $\pm 2\text{\AA}$. The method is basically interferometric, and the readings are cyclic with optical thickness. The quantity determined is optical thickness, and interpretation as physical thickness assumes knowledge of refractive index.

2. Application of Techniques to Backstreaming Tests

These two techniques were used to measure backstreaming rates by exposing specimen plates with known surface energies and optical properties to backstreaming vapors in a 36 inch diameter bell jar pumped by a 35 inch diffusion pump. Specimens were prepared by vacuum depositing coatings of nichrome and gold onto glass microscope slides. It was assumed that all oil molecules striking these surfaces would be physically adsorbed, up to monolayer coverage. A solenoid actuated shutter box was utilized to expose the specimens at various times during pump operation for intervals calculated to give partial coverage of the surfaces with condensed oil vapors. Eight specimens were exposed, 4 to DC 705 vapors and 4 to DC 704, for intervals ranging from 2 seconds to 5 1/4 hours at operating pressures from 3.2×10^{-6} torr to 1×10^{-7} torr. Both techniques were used on all specimens. In addition, two uncoated glass specimens were exposed to DC 704 vapors for contact angle measurements.

B. Backstreaming Tests-Test Series 1

For Test Series 1-1, the 35 inch pump was charged with 3 1/2 gallons of DC 705 fluid and operated at pressures below 1×10^{-6} torr to rid the pump of volatile vapors of residual cleaning fluid and light fractions of pump fluid. For each subsequent backstreaming test, the pump was cooled, the bell jar raised and the shutter box installed with a fresh specimen. The bell

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jar was then seated and the system roughed through the diffusion pump to a pressure below 50 microns before the diffusion pump was started. Approximately 20 minutes was required for roughing and from three to five hours were required to reach test pressure (approximately 1×10^{-6} torr), depending on previous exposure of the system to atmosphere.

For Test Series 1-2, the pump was drained, disassembled and cleaned before recharging with DC 704 fluid. The pump was operated before testing started to rid the system of volatile contaminants. In addition to the specimen in the shutter box, unshielded test specimens were held in comparable positions in the chamber, to determine the difference between steady state backstreaming and start-up and shut-down backstreaming.

Test 1-1 utilized gold coated microscope slides exclusively. Exposure times varied from 2 seconds at 9.7×10^{-7} to total exposure for 5 1/4 hours, including start-up and shut down. Four exposures were made. The slides were examined with the polarizing spectroscope and the contact angles of water (72 dynes/cm) and methylene iodide (50.7 dynes/cm) were determined. The critical surface tensions of the exposed slides were obtained by plots of the cosine of the contact angles against surface tension. Critical surface tensions were obtained for both unexposed slides and for slides wiped with silicone fluid. Results are given in Table 10.

The ellipsometric measurements indicated that no film was present on any of the slides. Contact angle measurements, which are less precise, tend to substantiate this. These plots are shown in Fig. 5. To check the apparatus, a gold slide was exposed to vapors in an unbaffled 2 inch diffusion pumped system which was known to backstream. Examination of this slide in the spectroscope showed the presence of a film, but no quantitative measurements could be made, due to the unevenness of the deposit. This slide exhibited a critical surface tension equal to that of the longest exposed slide in the original tests, and slightly less than that of the coated control slide. It is possible that a heavier film was present on all the slides during test, but which desorbed upon venting the chamber.

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TABLE 10
DATA FROM TEST SERIES 1-BACKSTREAMING TESTS

Slide No.	Slide Coating	Pump Fluid	Baffle Water	Baffle Temp. (°C)	Exposure Time	Exposure Pressure (torr)	Total Running Time (hrs.)	Critical Surface Tension (Dynes/cm)	Film Thickness by Ellipsometry (Å)
3	Gold	DC 705	Yes	10	2 sec.	9.7×10^{-7}	5 1/2	37	0
1	Gold	DC 705	Yes	10	2 min.	6×10^{-7}	6 3/4	(a)	0
2	Gold	DC 705	Yes	9	5 1/4 hrs.	$5.5 \times 10^{-2} - 3.2 \times 10^{-6}$	5 1/4	46	0
4	Gold	DC 705	No	16	4 3/4 hrs.	$2.0 \times 10^{-2} - 9.8 \times 10^{-7}$	4 3/4	42	0
Unused Gold								41	(a)
Unused Gold								40	(a)
Control Gold		DC 704			Film Applied by Wiping			49	(a)
10	Gold	Unknown	Exposed in Unbaffled 2" system	1 hr.		1×10^{-4}	1	45.5	Existence Verified - not Measurable
TEST 1-2									
22	Ni-Cr	DC 704	Yes	14	2 min.	1×10^{-6}	1 1/2	31	0
21	Ni-Cr	DC 704	Yes	14	1 1/2 hrs.	$2.4 \times 10^{-2} - 1 \times 10^{-6}$	1 1/2	34	0
11	Ni-Cr	DC 704	Yes	16	6 min.	1×10^{-7}	2	40	0
12	Ni-Cr	DC 704	Yes	16	2 hrs.	$3.0 \times 10^{-2} - 1 \times 10^{-6}$	2	43.5	50
Unused Ni-Cr								35.5	(a)
Control Ni-Cr		DC 704			Film Applied by Wiping			46.0	(a)
31	Glass	DC 704	Yes	14	1 1/2 hrs.	$24 \times 10^{-2} - 1 \times 10^{-6}$	1 1/2	81.5	(a)
32	Glass	DC 704	Yes	16	2 hrs.	$3.0 \times 10^{-2} - 1 \times 10^{-7}$	2	112	(a)
Unused Glass								73	(a)
Control Glass		DC 704			Film Applied by Wiping			103	(a)

(a) Not Measured

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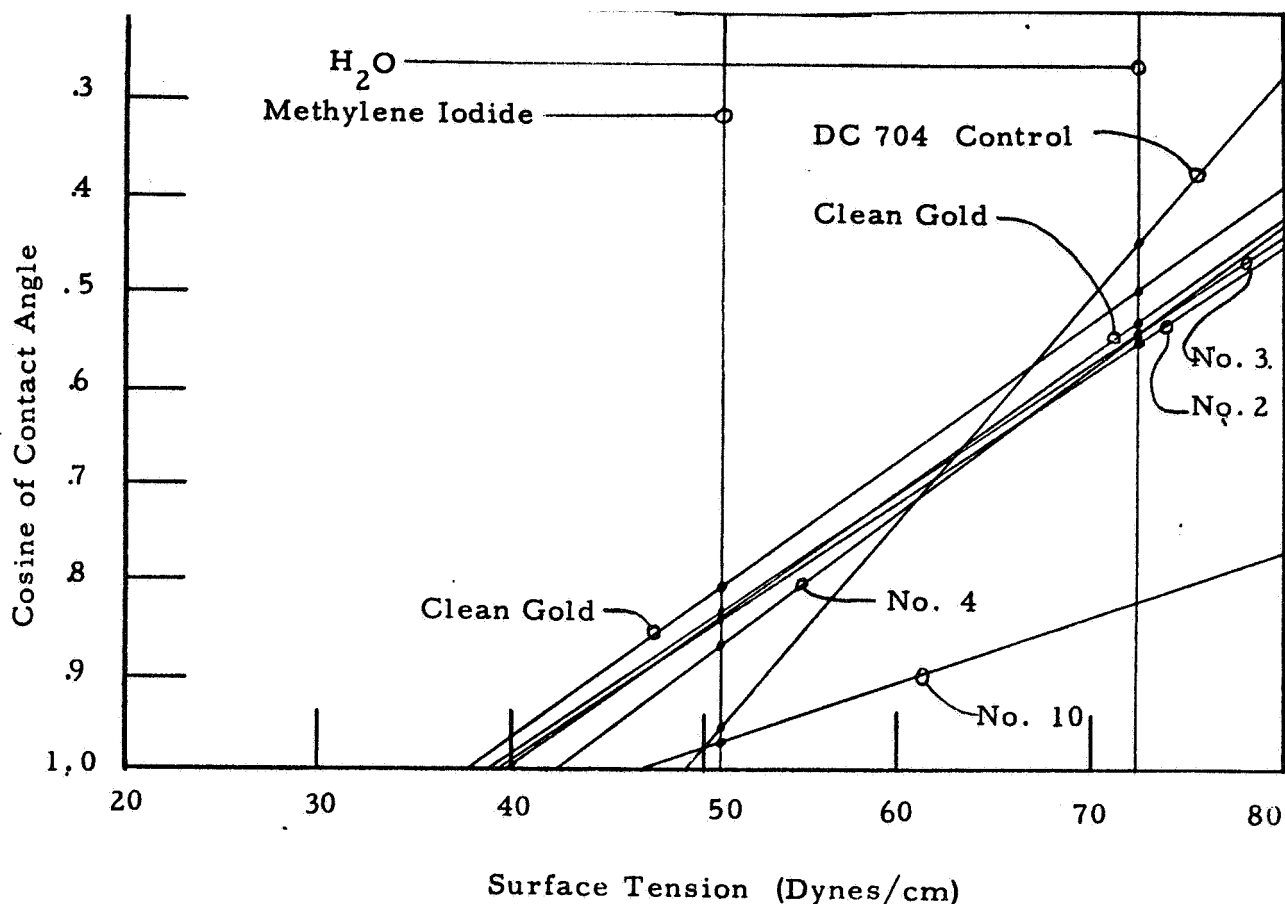


Fig. 5 CONTACT ANGLE MEASUREMENTS ON GOLD COATED GLASS SLIDES EXPOSED TO DC 705 VAPORS

Test 1-2, with DC-704 fluid, utilized nichrome coated glass slides and plain glass slides. It was thought that the more active metals would prevent desorption of the condensed film if any had occurred. The glass slides were included for control and were held in exposed positions outside the shutter box. These results also are presented in Table 10. Only the longest exposed slide showed any appreciable film by ellipsometry. This film may have accumulated during the rapid pump down procedure used in this test. This was measured to be 50 Å at about 1/2 hour after removal from the chamber, and desorbed to 37 Å after 60 hours exposure to room air. This slide exhibited the highest critical surface tension of any of the nichrome coated slides. The contact angle measurements on the glass slides were inconclusive. Plots of the contact angle measurements are shown in Fig. 6A and 6B.

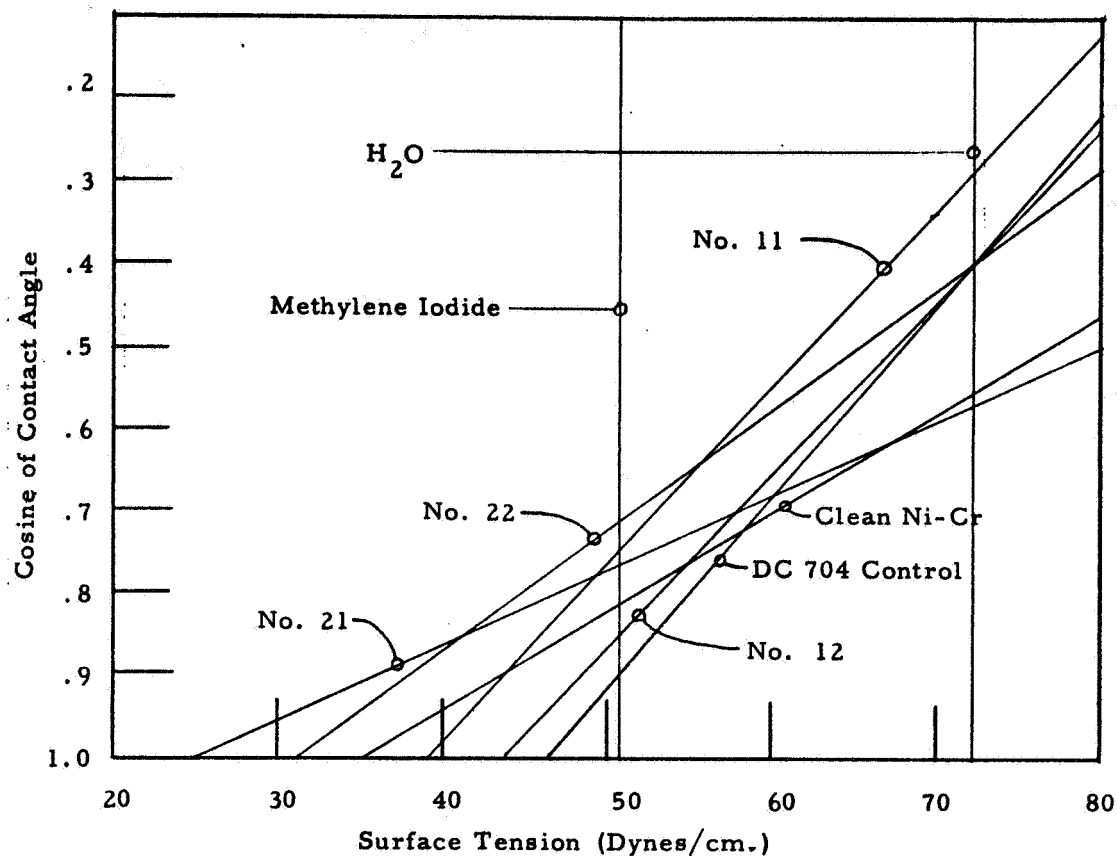


Fig. 6A CONTACT ANGLE MEASUREMENTS ON NICHROME COATED GLASS SLIDES EXPOSED TO DC 704 VAPORS

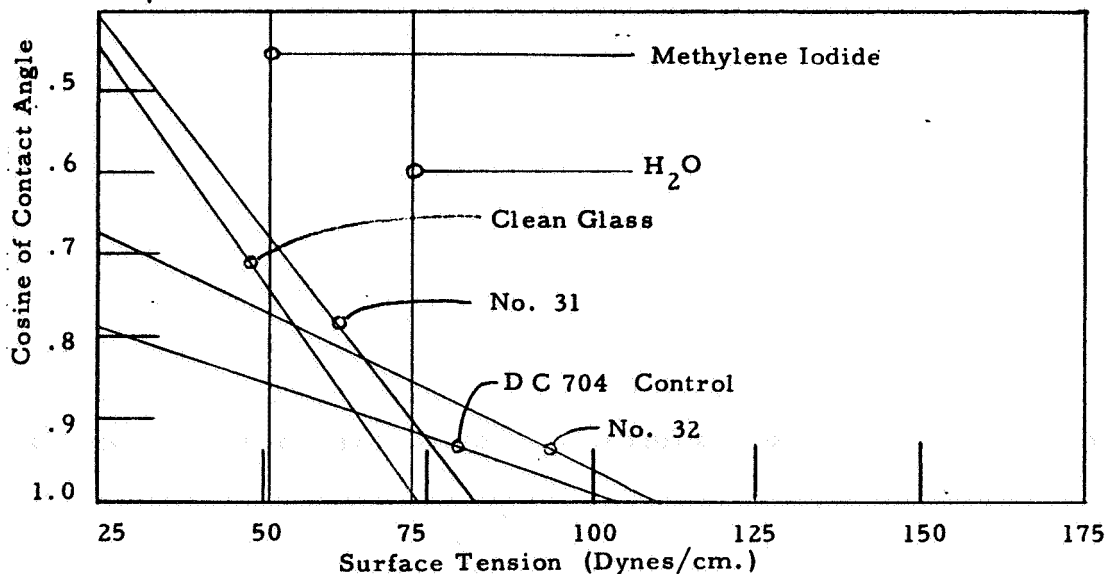


Fig. 6B CONTACT ANGLE MEASUREMENTS ON UNCOATED GLASS SLIDES EXPOSED TO DC 704 VAPORS

C. High Vacuum Testing Procedures - Test Series 2 and 3

Two vacuum systems were employed for two series of tests with various diffusion pump fluids. The apparatus is described in detail in Appendix B. A 6 inch diameter diffusion pumped system was used for initial testing of spontaneous and external ignition properties of three different pump fluids. A 35 inch diameter diffusion pumped system was used for spontaneous ignition tests with two of the pump fluids tested in the 6 inch pump. Initially, the tests were scheduled sequentially; the 35 inch pump tests were to be conducted after the termination of the 6 inch pump tests. Because of schedule slippage, the pump systems were operated independently and the test series overlapped.

Pump fluids which were selected for Test Series 2, in 6 inch pump systems, were Convalex 10, DC 705, and DC 704; and were tested in that order. Tests with the 6 inch pump system included a two week operation to establish system equilibrium, with subsequent admission of pure oxygen to 8 psia, observation for spontaneous combustion, and attempted ignition with automotive type spark plugs. Initial testing was accomplished after the two week operation according to the schedule below:

1. Connect oxygen admission system and sparking apparatus.
2. Purge oxygen inlet line between reservoir and bell jar.
3. Close foreline valve to mechanical pump.
4. Open chamber inlet valve and admit oxygen to chamber until 8 psia is indicated on gauge in inlet line.
5. Observe for spontaneous combustion as indicated by gross pressure rise.
6. Attempt ignition by sparking plugs in chamber, pump barrel, and foreline, in that order, with an automotive spark coil
7. If no combustion occurs reinitiate pumping by opening the foreline valve.

When combustion occurred in mechanical pump exhaust line after step 7 of test 2-1, this step was omitted in subsequent tests and the following step substituted.

8. If no combustion occurs, admit room atmosphere to the chamber and raise bell jar to purge system.

Subsequent to Test 2-1, the pump systems were equipped for remote testing from outside the building to insure the safety of test personnel.

For Test Series 3, with the 35 inch pump system, DC 705 and DC 704 pump fluids were selected and were tested in that order. Test sequencing was the same as for test series 2, with the omission of the attempted external spark ignition. Because of the size and length of the oxygen admission line, it was expedient to evacuate the line with a small mechanical vacuum pump to eliminate contaminant gases, rather than to purge it with oxygen.

1. Test Series 2 - Test Results

Test 2-1 was conducted on March 11 on the 6 inch system charged with Convalex 10 pump fluid, after 15 days of operation. The equilibrium pressure was approximately 8×10^{-7} torr as measured by the VG1A ionization gauge. Forepressure was maintained below 10 microns.

Preliminary to testing, the ionization and thermocouple gauges were turned off and the four bolts retaining the foreline spark plug flange were removed to vent explosive pressure rises if such occurred. The foreline vacuum held the unbolted flange in place with no leakage.

Oxygen was slowly admitted directly from a storage bottle through a steel fiber flame arrester and short length of tygon hose. Several minutes elapsed during admission. Approximately 2 minutes after the pressure had risen to the required level, the chamber spark plug was sparked several times with no observable effect. Approximately 2 minutes later the pump barrel spark plug was sparked and 5 minutes later the foreline plug was sparked. Observations continued for an additional two minutes with no observable combustion. At that time, the foreline valve to the mechanical pump was opened and pumping was restarted. Shortly thereafter, explosive combustion occurred in the exhaust duct of the mechanical pump. Pump operation was terminated and the resultant fire was extinguished immediately. Damage to the equipment was minor and did not delay subsequent testing appreciably.

After testing, room air was admitted to the vacuum chamber and the bell jar removed. White vapors were seen to be issuing from the pump throat, past the baffle. These vapors were noted on all subsequent tests

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and are concluded to be pump fluid vapors, since a heavy layer of condensed oil covered the base plate and bell jar interior. No quantitative determination was made of the amount of condensed oil found in the bell jar after test, but it was seen to be many orders of magnitude greater than could accumulate through backstreaming.

The oil vapor is attributed to continued application of heat in the pump boiler while the pump was at high pressure.

Efficient diffusion pump operation relies on supersonic flow of pump fluid vapors from the pump nozzles, which can occur only at pressures below about 10^{-2} torr. It is known that diffusion pumps can be stalled, and even made to run backwards (with respect to gas flow) under certain pressure conditions. It is highly probable that these conditions existed during testing and the resultant gas flow carried pump fluid vapors into the chamber.

Test 2-2 in the 6 inch pump system was conducted with DC 705 fluid. The system was then operated for 15 days at an equilibrium pressure of 1×10^{-6} torr and a forepressure of 10 microns or less.

On May 4, the system was tested as before with no observable combustion.

Test 2-3 was conducted in the 6 inch pump system with DC 704 pump fluid. The system was operated for 15 days at an equilibrium pressure of 9×10^{-7} torr with a forepressure below 8 microns.

Testing was accomplished as before with the substitution of a large polyethylene bag oxygen reservoir for the bottle and flame arrester. This substitution was made to increase the rate of oxygen admission at the request of the project sponsor's technical representative. The time to raise the pressure to 8 psia was thus reduced from 2 minutes to 45 seconds. No combustion was observed during the test.

2. Test Series 3 - Test Results

Test 3-1 was conducted in the 35 inch diameter diffusion pumped system with DC 705 pump fluid after the backstreaming tests of Test Series 1 were concluded.

The system was operated for 14 days at an equilibrium pressure of 8×10^{-8} torr with a forepressure of less than 10 microns.

On May 13, the system was tested as with the 6 inch pump system. The foreline valve was closed and the gauges turned off. Oxygen was admitted through a previously evacuated admission line from a polyethylene reservoir. One minute and ten seconds were required to raise the chamber pressure to 5 psia. At that time, the outlet from the reservoir became blocked with a fold in the bag. Oxygen flow was terminated for about 4 minutes while the outlet was freed. Flow was then reinitiated and the pressure rapidly increased to 8 psia. No combustion was observed and power to the pump heaters was disconnected and the system vented to atmosphere after a 15 minute "soak" at 8 psia.

Test 3-2 was conducted with DC 704 in the 35 inch pump system after the backstreaming tests were concluded. The system was operated for 14 1/2 days at an equilibrium pressure of 8×10^{-8} with a forepressure of 10 microns or less.

Testing was accomplished in the same manner as before with an oxygen admission time of 2 minutes to 8 psia. No combustion was observed after a 20 minute "soak" and the power was removed and the system vented to atmosphere.

3. Discussion of Results

The above reported results are summarized in Table 11. These substantiate the combustion test results which indicate that conditions normally encountered in diffusion pumped vacuum systems are not sufficiently severe to initiate or support combustion with DC 704, DC 705 or Convalex 10 pump fluids in oxygen rich atmospheres.

It should be recognized, however, that these tests evaluated only the immediate effects of the oxygen rich atmospheres. It has been observed by others that long term exposure to pure oxygen can convert normally incombustible materials into highly flammable substances. It is, therefore, recommended that vacuum systems utilizing pure oxygen atmospheres should not be operated after long term exposure until sufficient safety precautions are taken to insure incombustibility.

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TABLE 11
VACUUM CHAMBER COMBUSTION TEST RESULTS

Test No.	Test Fluid	Pump Size	Operation Time (days)	Equilibrium Pressure (torr)	Forepressure (Microns)	Combustion Occurrence			
						Spontaneous	Chamber Spark Plug Sparked	Pump Barrel Spark Plug Sparked	Foreline Spark Plug Sparked
2-1	Convalex 10	6"	15	8×10^{-7}	10	No	No	No	No
2-2	DC 705	6"	15	1×10^{-6}	10	No	No	No	No
2-3	DC 704	6"	15	9×10^{-7}	8	No	No	No	No
3-1	DC 705	35"	14	8×10^{-8}	10	No	--	--	--
3-2	DC 704	35"	14 1/2	8×10^{-8}	10	No	--	--	--

VII COMMENTS ON POINTS OF PARTICULAR INTEREST TO NASA

Poppet Valve- The installation of this valve is not considered necessary from the standpoint of explosion hazard, however, it is considered necessary so that: 1) the chamber may be isolated and 2) backstreaming may be eliminated at the initiation and shut-down of the diffusion pump and 3) admission of high pressure gases to the hot oils will not cause evolution of oil vapor into the chamber. Since the backstreaming rates at start-up and shut-down is reported to be orders of magnitude greater than the steady state value, and since our results show that the steady state value is very small, use of the poppet valve will reduce backstreaming to a negligible amount.

Heater Malfunction- The temperature of the oil in the diffusion pump is about 450° F during normal operation. We have stated in the conclusion that unless some outside source affects the reaction rate, the oil DC-705 should not explode below a pressure of 450 torr and a temperature of 600° F. Any heater malfunction should not allow the temperature to rise above this value.

Nitrogen Blanket- The part of emergency repressurization procedure which calls for injection of nitrogen into the diffusion pump throat is considered unnecessary from the stand point of diffusion pump fluid combustion hazards. The nitrogen will undoubtedly cause evolution of oil vapors past the pump throat as did the oxygen in the test conducted on this program. If a poppet valve is used, the injection is unnecessary. If the valve malfunctions, or if no valve is used, the oil vapors will enter the vacuum chamber where, under very specialized conditions, they may react with the oxygen at an ignition source, and may obscure vision by their density or by condensation on windows, visors or goggles. Since conditions within the diffusion pump will not normally cause combustion, the hazards of admitting oxygen directly to the pump will not be greater than those of admitting nitrogen.

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APPENDIX A

TABULATION OF EXPERIMENTAL COMBUSTION DATA

Some explanation is necessary to understand the information tabulated in this Appendix.

Definition of Terms:

Concentration Factor ϕ

$$\phi \equiv \frac{\text{Percent of Oil Present}}{\text{Percent of Stoichiometric Oil}}$$

$\phi > 1$, ϕ is written as $\frac{\phi}{\phi}$
 $\phi < 1$, ϕ is written as $\frac{\phi}{\phi}$
 E.G. $\phi = 2$ is written as $\frac{2}{2}$
 $\phi = \frac{1}{2}$ is written as $\frac{2}{2}$

The purpose of defining this quantity is to emphasize the equal importance of 100% excess air and 100% excess oil.

Explosion Very rapid rise in temperature and pressure producing a final pressure at least as great as the isothermal stoichiometric pressure increase,

SPI-Slight Pressure Increase Exhibited a slow pressure rise over a long period of time caused by either thermal decomposition or a slight reaction.

STI-Slight Temperature Increase Exhibits a rise in temperature not exceeding 12° F.

No Explosion No increase on either temperature or pressure was exhibited.

T res. Temperature of the reservoir

T cell Temperature of the cell

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TABLE I

SPONTANEOUS AND SPARK IGNITION DATA OF DC 705

Obtained in the new glass combustion cell

Stoichiometric Composition: 2.2% DC 705, 97.8% Oxygen

Nominal Experimental Conditions:

 $T_{res} = 288^{\circ}F$, $T_{cell} = 320^{\circ}F$

Reservoir Temperature, (°F)	Combustion Cell Temperature (°F)	Total Pressure (torr)	Concentra- tion Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli- joules)	Comments (Spark Ignition Experiment)
291	348	0.030	1.11	No explosion	6.76	No explosion
278	360	0.085	5.24	No explosion	6.76	No explosion
301	320	7.52	58.6	No explosion	3.00	No explosion
301	320	13.7	151.0	No explosion	3.00	No explosion
301	320	20.9	210.0	No explosion	3.00	No explosion
301	320	26.9	318.0	No explosion	3.96	No explosion
301	320	35.9	253.0	No explosion	3.96	No explosion
301	320	43.6	550.0	No explosion	3.96	No explosion
301	320	50.4	555.0	No explosion	3.96	No explosion
290	320	58.1	853.0	No explosion	3.96	No explosion
290	320	65.0	954.0	No explosion	3.96	No explosion
290	320	73.5	1079.0	No explosion	3.96	No explosion
290	320	83.8	1230.0	No explosion	3.96	No explosion
290	320	103.0	1510.0	No explosion	3.96	No explosion
290	320	120.0	1760.0	No explosion	3.96	No explosion
290	320	137.0	2010.0	No explosion	3.96	No explosion
290	320	163.0	2390.0	No explosion	3.96	No explosion
290	320	184.0	2700.	No explosion	3.96	No explosion
290	320	231.0	2950.	No explosion	3.96	No explosion
290	320	257.0	3770.	No explosion	3.96	No explosion
290	320	291.0	4260.	No explosion	3.96	No explosion
290	320	320.0	4700.	No explosion	3.96	No explosion
290	320	323.0	5760.	No explosion	3.96	No explosion
290	320	462.0	6760.	No explosion	5.95	No explosion
290	320	530.0	7780.	No explosion	5.95	No explosion
287	320	598.0	13150.	No explosion	5.95	No explosion

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A-1

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

T_{res} = 288°F, T_{cell} = 440

Reservoir Tempera- ture, (°F)	Combustion Cell Tempera- ture (°F)	Total Pressure (torr)	Concentra- tion Factor, Ø	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli- joules)	Comments (Spark Ignition Experiment)
314	451	0.030	3.03	No explosion	6.76	No explosion
310	418	0.074	1.05	No explosion	6.76	No explosion
290	444	0.094	2.06	No explosion	3.12	No explosion
290	444	3.84	84	No explosion	3.00	No explosion
290	444	5.46	120	No explosion	3.00	No explosion
290	444	8.55	188	No explosion	3.00	No explosion
290	445	17.5	385	No explosion	3.00	No explosion
290	445	30.8	678	No explosion	3.00	No explosion
290	445	37.6	826	No explosion	3.00	No explosion
290	445	46.2	1,015	No explosion	3.00	No explosion
290	445	53.	1,165	No explosion	3.00	No explosion
290	445	62.4	1,370	No explosion	3.00	No explosion
290	445	71.8	1,580	No explosion	3.00	No explosion
290	445	81.2	1,785	No explosion	3.00	No explosion
290	445	120	2,640	No explosion	3.00	No explosion
290	446	184	4,050	No explosion	3.00	No explosion
290	447	256	5,640	No explosion		
290	447	316	6,950	No explosion	5.8	No explosion
290	448	350	7,700	No explosion	5.8	No explosion
290	446	384	8,450	No explosion	5.8	No explosion
290	446	436	9,600	No explosion	5.8	No explosion
290	443	478	10,500	No explosion	5.8	No explosion
290	444	530	11,650	No explosion	5.8	No explosion
290	444	615	13,500	No explosion	5.8	No explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 288^{\circ}F$, $T_{cell} = 540^{\circ}F$

Reservoir Tempera- ture, ($^{\circ}F$)	Combustion Cell Tempera- ture ($^{\circ}F$)	Total Pressure (torr)	Concentra- tion Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli- joules)	Comments (Spark Ignition Experiment)
323	562	0.032	4.26	No explosion	6.76	No explosion
314	650	0.036	3.10	No explosion	6.76	No explosion
314	500	0.075	1.27	No explosion	6.76	No explosion
285	637	0.083	5.24	No explosion	6.76	No explosion
295	557	2.05	30.1	No explosion	5.8	No explosion
295	555	8.2	120	No explosion	5.8	No explosion
295	552	15.4	226	No explosion	5.8	No explosion
295	542	21.4	314	No explosion	5.8	No explosion
295	539	27.4	402	No explosion	4.76	No explosion
295	538	42.7	627	No explosion	4.76	No explosion
295	536	57.2	840	No explosion	4.76	No explosion
295	535	70	1,028	No explosion	4.76	No explosion
295	535	85.5	1,255	No explosion	4.76	No explosion
295	535	137	2,010	No explosion	4.76	No explosion
295	535	180	2,645	No explosion	4.76	No explosion
295	535	222	3,260	No explosion	4.76	No explosion
295	535	265	3,890	No explosion	4.76	No explosion
295	535	308	4,520	No explosion	4.76	No explosion
295	535	359	5,270	No explosion	4.76	No explosion
295	535	410	6,020	No explosion	4.76	No explosion
295	535	470	6,900	No explosion	4.75	No explosion
295	535	546	8,020	No explosion	4.75	No explosion
295	535	598	8,790	No explosion	4.75	No explosion

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Table 1 (Con't) DC 705 - Class Cell

Nominal Experimental Condition

 $T_{res} = 288^{\circ}F$, $T_{cell} = 800^{\circ}F$

Reservoir Temperature (°F)	Combustion Cell Temperature (°F)	Total Pressure (torr)	Concentra- tion Factor,	Combustion (Spontaneous Ignition Experiments	Spark Energy (Milli- joules)	Comments (Spark Ignition Experiment)
322	752	0.044	3.10	No Explosion	6.76	No Explosion
323	803	0.050	2.73	No Explosion	6.76	No Explosion
323	896	0.051	2.67	No Explosion	6.76	No Explosion
310	897	0.078	1.01	No Explosion	6.76	No Explosion
285	764	0.080	4.40	No Explosion	6.76	No Explosion
301	850	0.079	1.59	No Explosion	6.76	No Explosion
320	812	3.3	28.6	No Explosion	6.76	No Explosion
320	811	5.0	43.6	No Explosion	4.76	No Explosion
320	809	12.0	105.5	No Explosion	4.76	No Explosion
320	807	27.4	241	No Explosion	4.76	No Explosion
320	801	35.9	316	No Explosion	4.76	No Explosion
320	786	45.7	400	No Explosion	4.76	No Explosion
325	783	53.0	389	No Explosion	4.76	No Explosion
325	781	60.7	446	No Explosion	4.76	No Explosion
325	777	75.2	552	No Explosion	4.76	No Explosion
325	773	85.5	628	No Explosion	4.76	No Explosion
325	771	128.2	938	No Explosion	4.76	No Explosion
325	767	171.0	1,250	No Explosion	4.76	No Explosion
330	766	214.0	1,178	No Explosion	4.76	No Explosion
330	764	256.0	1,410	No Explosion	4.76	No Explosion
330	760	342.0	1,882	No Explosion	4.76	No Explosion
330	759	445.0	2,450	No Explosion	4.76	No Explosion
330	757	513.0	2,825	No Explosion	4.76	No Explosion
335	756	598.0	2,630	No Explosion	4.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

 $T_{res} = 288^{\circ}\text{F}$, $T_{cell} = 800^{\circ}\text{F}$

Reservoir Tempera- ture, ($^{\circ}\text{F}$)	Combustion Cell Tempera- ture ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentra- tion Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli- joules)	Comments (Spark Ignition Experiment)
371	373	0.85	1.23	No Explosion	6.76	No Explosion
371	377	3.4	3.24	No Explosion	6.76	No Explosion
371	377	5.1	4.89	No Explosion	6.76	No Explosion
372	382	6.8	6.29	No Explosion	6.76	No Explosion
372	386	8.5	7.90	No Explosion	6.76	No Explosion
372	395	0.068	1.60	No Explosion	6.76	No Explosion
386	422	6.85	4.02	No Explosion	6.76	No Explosion
386	422	12.8	7.52	No Explosion	6.76	No Explosion
386	422	21.4	12.6	No Explosion	6.76	No Explosion
386	422	30.03	17.6	No Explosion	6.76	No Explosion
386	422	36.8	21.6	No Explosion	6.76	No Explosion
386	422	44.5	26.2	No Explosion	6.76	No Explosion
386	422	57.4	33.7	No Explosion	6.76	No Explosion
386	422	65.0	38.2	No Explosion	6.76	No Explosion
386	422	72.8	42.8	No Explosion	6.76	No Explosion
386	422	80.5	47.3	No Explosion	6.76	No Explosion
357	422	102.5	60.2	No Explosion	6.76	No Explosion
357	422	120	70.5	No Explosion	6.76	No Explosion
357	422	137	80.5	No Explosion	6.76	No Explosion
357	422	154	90.5	No Explosion	6.76	No Explosion
357	422	171	100.5	No Explosion	6.76	No Explosion
357	422	205	121	No Explosion	6.76	No Explosion
357	422	222	131	No Explosion	6.76	No Explosion
368	422	256	150	No Explosion	6.76	No Explosion

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 390^{\circ}\text{F}$, $T_{cell} = 450^{\circ}\text{F}$

Reservoir Tempera- ture, ($^{\circ}\text{F}$)	Combustion Cell Tempera- ture ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentra- tion Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli- joules)	Comments (Spark Ignition Experiment)
364	449	1.7	2.32	No Explosion	6.76	No Explosion
364	453	3.4	4.64	No Explosion	6.76	No Explosion
364	453	5.1	6.97	No Explosion	6.76	No Explosion
364	448	6.8	9.29	No Explosion	6.76	No Explosion
364	440	0.69	1.06	No Explosion	6.76	No Explosion
391	445	5.47	3.21	No Explosion	6.76	No Explosion
391	445	5.47	3.21	No Explosion	6.76	No Explosion
391	445	17.1	10.0	No Explosion	6.76	No Explosion
391	445	27.4	16.1	No Explosion	6.76	No Explosion
391	445	41.0	24.0	No Explosion	6.76	No Explosion
391	445	51.3	30.1	No Explosion	6.76	No Explosion
391	445	63.3	37.1	No Explosion	6.76	No Explosion
391	445	72.7	42.6	No Explosion	6.76	No Explosion
391	445	83.8	49.2	No Explosion	6.76	No Explosion
391	445	102.5	64.4	No Explosion	6.76	No Explosion
391	445	120	75.3	No Explosion	6.76	No Explosion
391	445	137	86.0	No Explosion	6.76	No Explosion
391	445	154	96.5	No Explosion	6.76	No Explosion
391	445	171	107.2	No Explosion	6.76	No Explosion
391	445	231	145	No Explosion	6.76	No Explosion
391	445	248	156	No Explosion	6.76	No Explosion
382	445	274	172	No Explosion	6.76	No Explosion

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

 $T_{res} = 390^{\circ}\text{F}$, $T_{cell} = 500^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
375	495	5.55	4.88	No Explosion	6.76	No Explosion
375	495	18.8	16.5	No Explosion	6.76	No Explosion
375	495	25.6	22.5	No Explosion	6.76	No Explosion
375	495	35.9	31.6	No Explosion	6.67	No Explosion
375	495	47.0	41.3	No Explosion	6.67	No Explosion
375	495	58.0	51.0	No Explosion	6.76	No Explosion
375	495	66.6	58.6	No Explosion	6.67	No Explosion
375	495	77.0	67.6	No Explosio	6.67	No Explosion
375	495	85.5	75.2	No Explosi	6.76	No Explosion
375	495	102.5	90.0	No Explosion	6.76	No Explosion
375	495	119.5	105.0	No Explosion	6.76	No Explosion
375	495	137.0	121.0	No Explosion	6.76	No Explosion
375	495	154.0	135.0	No Explosion	6.76	No Explosion
375	495	171.0	150.0	No Explosion	6.76	No Explosion
375	495	189.0	162.0	No Explosion	6.76	No Explosion
375	495	201.0	177.0	No Explosion	6.76	No Explosion
375	495	214.0	188.0	No Explosion	6.76	No Explosion
375	495	231.0	203.0	No Explosion	6.76	No Explosion
375	495	239	239	No Explosion	6.76	No Explosion
375	495	266	266	No Explosion	6.76	No Explosion
369	495	274	274	No Explosion	6.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions.

T_{res} = 390°F T_{cell} = 550°F

Reservoir Temperature, (°F)	Combustion Cell Temperature (°F)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
360	551	2.00	3.3	No Explosion	6.76	No Explosion
360	552	3.40	5.6	No Explosion	6.76	No Explosion
361	550	4.30	6.7	No Explosion	6.76	No Explosion
361	545	6.80	10.5	No Explosion	6.76	No Explosion
362	541	8.00	12.2	No Explosion	6.76	No Explosion
363	523	0.07	10.4	No Explosion	6.76	No Explosion
369	545	3.34	3.68	No Explosion	6.76	No Explosion
369	545	12.0	13.2	No Explosion	6.76	No Explosion
369	545	19.7	21.7	No Explosion	6.76	No Explosion
369	545	25.6	28.2	No Explosion	6.76	No Explosion
369	545	34.2	37.6	No Explosion	6.76	No Explosion
369	545	46.2	50.8	No Explosion	6.76	No Explosion
369	545	54.7	60.3	No Explosion	6.76	No Explosion
369	545	58.2	64.2	No Explosion	6.76	No Explosion
369	545	66.6	73.4	No Explosion	6.76	No Explosion
369	545	73.5	81.0	No Explosion	6.76	No Explosion
369	545	80.5	88.6	No Explosion	6.76	No Explosion
369	545	102.5	113	No Explosion	6.76	No Explosion
369	545	111.0	122	No Explosion	6.76	No Explosion
369	545	128.0	141	No Explosion	6.76	No Explosion
369	545	145.0	160	No Explosion	6.76	No Explosion
369	545	163.0	179	No Explosion	6.76	No Explosion
369	545	184.0	203	No Explosion	6.76	No Explosion
369	545	197.0	217	No Explosion	6.76	No Explosion
369	545	214.0	236	No Explosion	6.76	No Explosion
369	545	231.0	254	No Explosion	6.76	No Explosion
369	545	248.0	273	No Explosion	6.76	No Explosion
369	545	256.0	282	No Explosion	6.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 390^{\circ}\text{F}$ $T_{cell} = 620^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
365	641	1.30	1.69	No Explosion	6.76	No Explosion
363	643	2.40	3.4	No Explosion	6.76	No Explosion
360	631	3.60	5.9	No Explosion	6.76	No Explosion
360	632	4.87	8.0	No Explosion	6.76	No Explosion
360	645	5.98	9.7	No Explosion	6.76	No Explosion
360	637	8.12	13.30	No Explosion	6.76	No Explosion
359	632	0.07	8.45	No Explosion	6.76	No Explosion
370	620	0.17	5.9	No Explosion	6.76	No Explosion
370	620	2.74	2.74	No Explosion	6.76	No Explosion
370	620	6.15	6.15	No Explosion	6.76	No Explosion
370	620	13.6	13.6	No Explosion	6.76	No Explosion
370	620	22.2	22.2	No Explosion	6.76	No Explosion
370	620	30.0	30.0	No Explosion	6.76	No Explosion
370	620	41.0	41.0	No Explosion	6.76	No Explosion
370	620	45.3	45.3	No Explosion	6.76	No Explosion
370	620	54.6	54.6	No Explosion	6.76	No Explosion
370	620	62.4	62.4	No Explosion	6.76	No Explosion
370	620	70.0	70.0	No Explosion	6.76	No Explosion
370	620	81.2	81.2	No Explosion	6.76	No Explosion
370	620	102.5	102.5	No Explosion	6.76	No Explosion
370	620	128	128	No Explosion	6.76	No Explosion
370	620	179	179	No Explosion	6.76	No Explosion
370	620	214	214	No Explosion	6.76	No Explosion
370	620	265	265	No Explosion	6.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 390^{\circ}\text{F}$ $T_{cell} = 700^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
368	744	1.4	1.62	No Explosion	6.76	No Explosion
367	745	2.4	2.88	No Explosion	6.76	No Explosion
367	744	4.7	5.63	No Explosion	6.76	No Explosion
367	749	5.8	6.94	No Explosion	6.76	No Explosion
367	752	7.7	9.20	No Explosion	6.76	No Explosion
367	748	0.07	12.00	No Explosion	6.76	No Explosion
372	710	2.01	2.67	No Explosion	6.76	No Explosion
372	710	2.91	2.67	No Explosion	6.76	No Explosion
372	710	10.2	9.35	No Explosion	6.76	No Explosion
372	710	22/3	20.45	No Explosion	6.76	No Explosion
372	710	34.2	31.4	No Explosion	6.76	No Explosion
372	710	42.8	39.3	No Explosion	6.76	No Explosion
372	710	54.0	49.5	No Explosion	6.76	No Explosion
372	710	60.0	55.0	No Explosion	6.76	No Explosion
372	710	68.5	63.1	No Explosion	6.76	No Explosion
372	710	78.7	72.3	No Explosion	6.76	No Explosion
372	710	90.0	82.6	No Explosion	6.76	No Explosion
372	710	128	117.5	No Explosion	6.76	No Explosion
372	710	180	165	No Explosion	6.76	No Explosion
372	710	214	1965	No Explosion	6.76	No Explosion
372	710	264	243	No Explosion	6.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 390^{\circ}\text{F}$ $T_{cell} = 800^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
380	859	1.4	1.02	No Explosion		No Explosion
378	854	1.9	1.50	No Explosion	6.76	No Explosion
377	854	3.5	2.93	No Explosion	6.76	No Explosion
377	850	5.4	4.40	No Explosion	6.76	No Explosion
375	842	6.6	5.76	No Explosion	6.76	No Explosion
373	841	8.5	7.98	No Explosion	6.76	No Explosion
368	829	0.07	12.3	No Explosion	6.76	No Explosion
376	810	3.25	2.65	No Explosion	6.76	No Explosion
376	810	17.1	13.9	No Explosion	6.76	No Explosion
376	810	29.1	23.7	No Explosion	6.76	No Explosion
376	810	42.0	34.2	No Explosion	6.76	No Explosion
376	810	56.5	46.0	No Explosion	6.76	No Explosion
376	810	65.0	53.0	No Explosion	6.76	No Explosion
376	810	72.7	59.3	No Explosion	6.76	No Explosion
376	810	94.0	76.7	No Explosion	6.76	No Explosion
376	810	137	112	No Explosion	6.76	No Explosion
376	810	163	133	No Explosion	6.76	No Explosion
376	810	205	167	No Explosion	6.76	No Explosion
376	810	274	223	No Explosion	6.76	No Explosion

Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 390^{\circ}\text{F}$ $T_{cell} = 900^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
381	900	8.55	7.85	No Explosion	6.76	No Explosion
381	895	15.4	12.55	No Explosion	6.76	No Explosion
381	890	29.1	21.3	No Explosion	6.76	No Explosion
381	892	34.2	22.8	No Explosion	6.76	No Explosion
381	894	42.7	26.1	No Explosion	6.76	No Explosion
381	896	60.0	33.8	No Explosion	6.76	No Explosion
381	898	68.5	36.8	No Explosion	6.76	No Explosion
381	900	75.2	37.7	No Explosion	6.76	No Explosion
381	901	85.5	39.2	No Explosion	6.76	No Explosion
381	902	137	60.3	No Explosion	6.76	No Explosion
381	903	180	76.2	No Explosion	6.76	No Explosion
381	904	222	92.2	No Explosion	6.76	No Explosion
381	905	265	108	No Explosion	6.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

T_{res} = 440°F T_{cell} = 460°F

Reservoir Tempera- ture, (°F)	Combustion Cell Tempera- ture (°F)	Total Pressure (torr)	Concentra- tion Factor, Ø	Comments (Spontaneous Ignition Experiment)	Spark Energy (mJoules)	Comments (Spark Ignition Experiment)
443	453	4.45	<u>3.83</u>	No Explosion	4.76	No Explosion
443	453	6.66	<u>2.55</u>	No Explosion	4.76	No Explosion
443	453	8.12	<u>2.10</u>	No Explosion	4.76	No Explosion
443	460	10.2	<u>1.67</u>	No Explosion	4.76	No Explosion
443	460	12.0	<u>1.42</u>	No Explosion	4.76	No Explosion
443	460	16.2	<u>1.05</u>	No Explosion	4.76	No Explosion
443	460	20.5	1.20	No Explosion	4.76	No Explosion
443	460	23.9	1.40	No Explosion	4.76	No Explosion
443	460	29.1	1.70	No Explosion	4.76	No Explosion
441	460	30.8	1.78	No Explosion	4.76	No Explosion
441	460	33.5	1.94	No Explosion	4.76	No Explosion
441	460	57.3	3.31	No Explosion	4.76	No Explosion
441	460	77.8	4.49	No Explosion	4.76	No Explosion
441	460	115.0	6.65	No Explosion	4.76	No Explosion
441	460	188.0	10.85	No Explosion	4.76	No Explosion
441	465	350.0	20.2	No Explosion	4.76	No Explosion
441	465	163.0	9.4	No Explosion	4.76	No Explosion
441	465	80.5	<u>4.65</u>	No Explosion	4.76	No Explosion
441	465	75.5	<u>4.36</u>	No Explosion	4.76	No Explosion
441	465	32.5	1.88	No Explosion	4.76	No Explosion
441	465	24.0	1.38	No Explosion	4.76	No Explosion
441	465	15.4	<u>1.12</u>	No Explosion	4.76	No Explosion
441	465	6.34	<u>2.74</u>	No Explosion	4.76	No Explosion
441	465	3.42	<u>5.09</u>	No Explosion	4.76	No Explosion
440	467	0.68	26.7	No Explosion	4.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

T_{res} = 440°F T_{cell} = 530°F

Reservoir Temperature, (°F)	Combustion Cell Temperature (°F)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
440	525	4.8	<u>2.84</u>	No Explosion	4.76	No Explosion
440	525	5.3	<u>4.29</u>	No Explosion	4.76	No Explosion
440	525	12.0	<u>2.65</u>	No Explosion	4.76	No Explosion
440	525	20.5	<u>1.77</u>	No Explosion	4.76	No Explosion
488	525	30.0	<u>1.52</u>	No Explosion	4.76	No Explosion
488	539	42.0	<u>1.19</u>	No Explosion	4.76	No Explosion
488	539	53.0	<u>1.03</u>	No Explosion	4.76	No Explosion
488	539	98.5	1.67	No Explosion	4.76	No Explosion
488	539	154.0	2.5	No Explosion	4.76	No Explosion
488	539	256.0	4.02	No Explosion	4.76	No Explosion
505	539	342.0	5.00	Pump down	4.76	Pump down
505	539	163.0	2.56	No Explosion	4.76	No Explosion
505	539	85.5	1.45	No Explosion	4.76	No Explosion
505	539	34.2	<u>1.6</u>	No Explosion	4.76	No Explosion
505	539	15.8	<u>2.88</u>	No Explosion	4.76	No Explosion
505	539	7.7	5.32	No Explosion	4.76	No Explosion
505	539	1.37	<u>26.5</u>	No Explosion	4.76	No Explosion
468	546	8.05	<u>3.96</u>	No Explosion	4.76	No Explosion

Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

T_{res} = 440°F, T_{cell} = 620°F

Reservoir Temperature, (°F)	Combustion Cell Temperature (°F)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
475	623	1.02	<u>35</u>	No Explosion	4.76	No Explosion
475	623	3.42	<u>9.95</u>	No Explosion	4.76	No Explosion
475	623	6.15	<u>5.91</u>	No Explosion	4.76	No Explosion
475	623	17.9	<u>2.16</u>	No Explosion	4.76	No Explosion
475	623	30.8	<u>1.33</u>	No Explosion	4.76	No Explosion
475	623	41.0	<u>1.05</u>	No Explosion	4.76	No Explosion
475	623	65.9	1.45	No Explosion	4.76	No Explosion
475	623	85.5	1.78	No Explosion	4.76	No Explosion
475	623	179.0	3.57	No Explosion	4.76	No Explosion
475	623	269.0	5.36	No Explosion	4.76	No Explosion
495	623	350.0	6.7	Pump down	4.76	Pump down
495	623	154.0	2.95	No Explosion	4.76	No Explosion
495	623	66.7	1.32	No Explosion	4.76	No Explosion
495	623	34.2	<u>1.46</u>	No Explosion	4.76	No Explosion
495	623	23.1	<u>1.97</u>	No Explosion	4.76	No Explosion
495	623	17.9	<u>2.54</u>	No Explosion	4.76	No Explosion
495	623	9.4	<u>4.60</u>	No Explosion	4.76	No Explosion
483	623	2.05	<u>20.0</u>	No Explosion	4.76	No Explosion

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Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 440^{\circ}\text{F}$ $T_{cell} = 740^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
453	740	1.37	<u>16.6</u>	No Explosion	4.76	No Explosion
453	740	2.82	<u>16.1</u>	No Explosion	4.76	No Explosion
435	740	10.3	<u>6.64</u>	No Explosion	5.2	No Explosion
435	740	19.7	<u>4.61</u>	No Explosion	5.2	No Explosion
435	740	30.8	<u>3.69</u>	No Explosion	5.2	No Explosion
435	740	38.5	<u>3.53</u>	No Explosion	5.2	No Explosion
435	740	46.2	<u>3.44</u>	No Explosion	5.2	No Explosion
435	740	70.2	<u>1.18</u>	No Explosion	5.2	An Explosion
545	740	74.5	2.74	No Explosion	5.2	An Explosion
482	735	20.5	1.99	No Explosion	10.2	An Explosion

Nominal Experimental Conditions:
 $T_{res} = 440^{\circ}\text{F}$ $T_{cell} = 750^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
444	745	4.02	4.52	No Explosion	6.76	No Explosion
444	745	10.25	<u>2.44</u>	No Explosion	6.76	No Explosion
444	745	18.8	<u>1.69</u>	No Explosion	6.76	No Explosion
444	745	25.6	<u>1.51</u>	No Explosion	6.76	An Explosion
485	745	29.0	<u>1.51</u>	No Explosion	6.76	An Explosion
506	735	24.0	2.84	No Explosion	6.76	No Explosion

Nominal Experimental Conditions:
 $T_{res} = 440^{\circ}\text{F}$ $T_{cell} = 800^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
470	780	2.05	<u>15.5</u>	No Explosion	3.96-6.76	No Explosion
470	790	2.56	<u>12.4</u>	No Explosion	3.96-6.76	No Explosion
470	790	2.99	<u>10.65</u>	No Explosion	3.96-6.76	No Explosion
470	790	3.60	<u>8.85</u>	No Explosion	3.96-6.76	No Explosion
470	790	4.00	<u>7.95</u>	No Explosion	3.96-6.76	No Explosion
470	790	19.7	<u>1.62</u>	No Explosion	3.96-6.76	No Explosion
470	790	22.2	<u>1.64</u>	No Explosion	3.96-6.76	No Explosion
470	790	26.0	<u>1.40</u>	No Explosion	3.96-6.76	No Explosion
470	790	29.0	<u>1.25</u>	No Explosion	3.96-6.76	No Explosion
470	790	31.6	<u>1.15</u>	No Explosion	3.96-6.76	No Explosion
470	790	34.2	<u>1.05</u>	No Explosion	3.96-6.76	No Explosion
470	790	41.0	<u>1.00</u>	No Explosion	3.96-6.76	No Explosion
470	790	47.0	1.15	No Explosion	3.96-6.76	No Explosion
470	790	56.5	1.37	No Explosion	3.96-6.76	No Explosion
470	790	72.0	1.76	No Explosion	3.96-6.76	No Explosion
470	790	85.5	2.09	No Explosion	3.96-6.76	No Explosion
470	790	180.0	4.4	No Explosion	3.96-6.76	No Explosion
490	790	270.0	5.94	No Explosion	3.96-6.76	No Explosion

Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:

 $T_{res} = 440^{\circ}\text{F}$ $T_{cell} = 800^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
470	800	8.55	3.72	No Explosion	3.96-6.76	No Explosion
470	800	17.1	1.86	No Explosion	3.96-6.76	No Explosion
470	800	20.5	1.55	No Explosion	3.96-6.76	No Explosion
470	800	25.6	1.27	No Explosion	3.96-6.76	No Explosion
470	800	33.3	1.05	No Explosion	3.96-6.76	No Explosion
470	800	20.5	1.55	No Explosion	3.96-6.76	No Explosion
470	800	29.9	1.06	No Explosion	3.96	An Explosion

Nominal Experimental Conditions:

 $T_{res} = 440^{\circ}\text{F}$ $T_{cell} = 850^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
480	854	2.82	12.9	No Explosion	3.8-6.76	No Explosion
480	854	9.4	5.32	No Explosion	3.8-6.76	No Explosion
480	854	18.8	3.39	No Explosion	3.8-6.76	No Explosion
480	854	26.5	2.92	No Explosion	3.8-6.76	No Explosion
480	854	34.2	2.92	No Explosion	3.8-6.76	No Explosion
480	854	51.3	2.04	No Explosion	3.8-6.76	No Explosion
480	854	80.5	1.47	No Explosion	3.8-6.76	No Explosion
480	854	179.0	1.36	No Explosion	3.8-6.76	No Explosion
530	854	308.0	2.18	No Explosion	3.8-6.76	No Explosion

Nominal Experimental Conditions:

 $T_{res} = 570^{\circ}\text{F}$ $T_{cell} = 630^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
563	630	8.55	6.59	No Explosion	6.76	No Explosion
563	630	17.1	7.3	No Explosion	6.76	No Explosion
563	630	27.4	7.05	No Explosion	6.76	No Explosion
563	630	32.5	8.05	No Explosion	6.76	No Explosion
563	630	35.9	9.18	No Explosion	6.76	No Explosion
563	630	46.2	8.63	No Explosion	6.76	No Explosion
563	630	56.5	8.30	No Explosion	6.76	No Explosion
593	630	65.0	8.21	No Explosion	6.76	No Explosion
593	630	73.5	9.57	No Explosion	6.76	An Explosion
593	630	102.0		No Explosion	6.76	An Explosion

Nominal Experimental Conditions:

 $T_{res} = 570^{\circ}\text{F}$ $T_{cell} = 715^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
560	715	17.1	17.3	No Explosion	6.76	No Explosion
560	715	27.4	19.9	No Explosion	6.76	No Explosion
560	715	35.8	22.8	No Explosion	6.76	No Explosion
560	715	46.2	23.6	No Explosion	6.76	No Explosion
560	715	55.5	24.6	No Explosion	6.76	No Explosion
560	715	65.0	24.5	No Explosion	6.76	No Explosion
634	715	73.5	24.7	No Explosion	6.76	An Explosion

Table 1 (Con't) DC 705 - Glass Cell

Nominal Experimental Conditions:
 $T_{res} = 570^{\circ}\text{F}$ $T_{cell} = 800^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
515	800	4.1	<u>22.7</u>	No Explosion	6.76	No Explosion
515	800	11.1	<u>34.9</u>	No Explosion	6.76	No Explosion
515	800	18.8	<u>36.3</u>	No Explosion	6.76	No Explosion
515	800	29.1	<u>34.2</u>	No Explosion	6.76	No Explosion
515	800	39.4	<u>32.3</u>	No Explosion	6.76	No Explosion
619	790	48.8	<u>36.3</u>	No Explosion	6.76	An Explosion

Nominal Experimental Conditions:
 $T_{res} = 570^{\circ}\text{F}$ $T_{cell} = 850^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
580	850	8.55	<u>58.8</u>	No Explosion	6.76	No Explosion
580	850	19.7	<u>25.4</u>	No Explosion	6.76	No Explosion
580	850	29.9	<u>16.7</u>	No Explosion	6.76	No Explosion
580	850	37.6	<u>13.3</u>	No Explosion	6.76	An Explosion
580	850	50.0		No Explosion	6.76	No Explosion

Nominal Experimental Conditions:
 $T_{res} = 570^{\circ}\text{F}$ $T_{cell} = 850^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
530	850	6.67	<u>21.1</u>	No Explosion	3.8-6.76	No Explosion
530	850	19.7	<u>9.25</u>	No Explosion	3.8-6.76	No Explosion
530	850	25.6	<u>8.88</u>	No Explosion	3.8-6.76	No Explosion
530	850	37.6	<u>7.85</u>	No Explosion	3.8-6.76	An Explosion
560	850	25.6	11.5	No Explosion	3.8	An Explosion

Nominal Experimental Conditions:
 $T_{res} = 570^{\circ}\text{F}$ $T_{cell} = 900^{\circ}\text{F}$

Reservoir Temperature, ($^{\circ}\text{F}$)	Combustion Cell Temperature ($^{\circ}\text{F}$)	Total Pressure (torr)	Concentration Factor, ϕ	Comments (Spontaneous Ignition Experiment)	Spark Energy (milli-joules)	Comments (Spark Ignition Experiment)
510	900	4.36	<u>17.7</u>	No Explosion	6.76	No Explosion
510	900	12.0	<u>9.85</u>	No Explosion	6.76	No Explosion
510	900	15.4	<u>10.4</u>	No Explosion	6.76	No Explosion
510	900	18.8	<u>10.9</u>	No Explosion	6.76	No Explosion
510	900	25.6	<u>9.75</u>	No Explosion	6.76	No Explosion
560	890	35.0	<u>8.45</u>	No Explosion	6.76	An Explosion

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Table 1 (con't) DC 705-Glass Cell

Nominal Experimental Conditions

The cell is initially filled to 256 torr of oxygen at 752°F and the temperature of the cell is raised until the vapor pressure exceeds 100 torr

Reservoir Temperature (°F)	Combustion cell temperature (°F)	Total Pressure (torr)	Pressure of DC 705 (torr)	Concentration Factor	Comments
426	752	256	0.78	2.81	No Explosion
602	744	258	0.81	2.71	No Explosion
654	744	265	2.22	1.10	No Explosion
708	744	270	7.42	3.37	No Explosion
756	748	279	17.90	12.29	No Explosion
799	752	282	39.70	18.05	No Explosion
799	756	316	A large amount of thermal decomposition is occurring in this region but no explosion has occurred.		Thermal Decomposition
799	752	325			

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TABLE 2
SPONTANEOUS IGNITION DATA OF DC-705 OBTAINED
IN THE STAINLESS STEEL COMBUSTION CELL

Stoichiometric Mixture: 2.2% D. C. 705, 97.8% Oxygen

Nominal Experimental Conditions:

T_{Res.} = 540°F; T_{Cell} = 580°F

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (Torr)	Comments
544	580	10	No Explosion
544	579	25	No Explosion
543	578	36	No Explosion
543	577	43	No Explosion
543	577	60	No Explosion
543	577	74	No Explosion
543	577	91	No Explosion
543	577	100	No Explosion
543	577	120	No Explosion
541	577	140	No Explosion
541	577	160	No Explosion
540	577	180	S. T. I.
540	579	200	No Explosion
540	579	220	No Explosion
540	582	240	No Explosion
540	582	260	No Explosion
540	585	400	No Explosion
540	587	480	No Explosion
540	587	520	No Explosion
540	587	700	No Explosion
540	588	780	No Explosion
540	575	7	No Explosion
540	585	110	No Explosion
540	580	170	No Explosion
540	580	240	No Explosion
540	582	330	No Explosion
540	583	460	No Explosion
540	585	640	S. P. I.
540	586	720	No Explosion
540	592	820	No Explosion

Table 2 (Con't) DC 705-Stainless Steel Cell
 Nominal Experimental Conditions
 T_{res} = 530°F, T_{cell} = 720°F

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
546	710	36	No Explosion
544	708	43	No Explosion
543	707	44	No Explosion
541	711	58	No Explosion
540	710	68	No Explosion
538	713	80	No Explosion
537	713	95	No Explosion

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Table 2 (Con't) DC 705-Stainless Steel Cell
Nominal Experimental Conditions
T_{res} = 530°F, T_{cell} = 720°F

Reservoir Temp. °F	Combustion Cell Temp. °F	Total Pressure torr	Comments
535.5	718	115	No Explosion
534.	716	150	No Explosion
532.5	720	180	No Explosion
531	721	242	No Explosion
529.5	722	290	No Explosion
528	709	295	No Explosion
526.5	720	342	No Explosion
525	720	350	No Explosion
523.5	726	400	No Explosion
522	726	440	No Explosion
520.5	728	495	No Explosion
519	728	545	No Explosion
517.5	730	605	No Explosion
516	731	650	No Explosion
514.5	731	700	No Explosion
514	733	745	No Explosion
513	734	800	No Explosion

Table 2 (Con't) DC 705-Stainless Steel Cell
Nominal Experimental Conditions
T_{res} = 515°F T_{cell} = 810°F

Reservoir Temp. °F	Combustion Cell Temp. °F	Total Pressure torr	Comments
516	798	24	S.P.I
516	796	25	S.P.I
516	800	32.5	S.P.I
516	797	33	S.P.I
516	801	44	S.P.I
516	800	54.5	S.P.I
516	802	69.5	S.P.I
516	802	84	S.P.I
516	798	86	S.P.I
516	798	95	
516	800	105	No Explosion
516	808	145	No Explosion
516	802	180	No Explosion
519	808	235	No Explosion
519	809	295	No Explosion
519	809	340	No Explosion
519	811	400	No Explosion
519	811	460	No Explosion
519	811	490	No Explosion
519	813	545	No Explosion
519	815	600	No Explosion
519	820	700	No Explosion
519	817	740	No Explosion
514	820	800	No Explosion

Table 2 (Con't) DC 705-Stainless Steel Cell
Nominal Experimental Conditions
T_{res} = 560°F T_{cell} = 905°F

Reservoir Temp. °F	Combustion Cell Temp. °F	Total Pressure torr	Comments
535	900	12	No Explosion
535	908	29	No Explosion
535	900	28	No Explosion
535	901	34.5	No Explosion
567	904	48	S.P.I
567	903	66	S.P.I
567	899	74	No Explosion
567	904	95.5	No Explosion

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Table 2 (Con't) DC 705-Stainless Steel Cell
Nominal Experimental Conditions
T_{res} = 560°F T_{cell} = 905°F

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
567	899	96	No Explosion
567	899	97	No Explosion
567	906	140	No Explosion
567	900	180	No Explosion
567	902	220	No Explosion
567	902	250	No Explosion
567	901	255	No Explosion
567	905	300	No Explosion
567	906	330	No Explosion
567	910	390	No Explosion
567	905	415	No Explosion
567	909	460	No Explosion
567	908	490	No Explosion
567	907	550	No Explosion
567	912	650	No Explosion
567	915	700	No Explosion
585	918	800	No Explosion

Table 2 (con't) DC 705-Stainless Steel Cell
Nominal Experimental Conditions

The cell is initially filled to 344 torr of oxygen at 476°F and the temperature of the cell is raised until the vapor pressure exceeds 100 torr

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
476	704	344	No Explosion
492	704	344	No Explosion
498	704	344	No Explosion
528	702	368	No Explosion
578	703	368	No Explosion
628	704	393	No Explosion
670	704	417	No Explosion
674	704	417	No Explosion
680	704	417	No Explosion
693	704	417	No Explosion
716	704	417	No Explosion
722	704	442	No Explosion
735	704	442	No Explosion
743	704	442	No Explosion
766	704	442	No Explosion

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TABLE 3

SPONTANEOUS IGNITION DATA OF CONVALEX 10 IN THE
STAINLESS STEEL COMBUSTION CELL

Stoichiometric Mixture: 2.9% Convalex 10, 97.1% Oxygen

Nominal Experimental Conditions:

$T_{res} = 650^{\circ}\text{F}$, $T_{cell} = 700^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
687	700	20	No Explosion
684	694	263	No Explosion
683	696	393	No Explosion
682	696	513	No Explosion
680	696	703	S.T.I.
677	696	3.5	S.T.I.
671	696	505	S.T.I. & S.P.I.
667	696	403	S.T.I. & S.P.I.
665	696	263	S.T.I. & S.P.I.
660	696	115	S.T.I. & S.P.I.
658	696	75	S.T.I. & S.P.I.
656	697	10	No Explosion

Table 3 (con't) Convalex 10-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 680^{\circ}\text{F}$ $T_{cell} = 800^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
688	796	122	No Explosion
687	798	345	No Explosion
687	799	373	No Explosion
686	802	495	No Explosion
686	805	648	No Explosion
685	806	755	No Explosion
685	805	755	No Explosion
685	805	755	No Explosion
685	804	665	No Explosion
685	802	550	No Explosion
685	799	428	No Explosion
685	797	305	No Explosion
685	795	200	No Explosion
685	794	97	No Explosion
685	794	85	No Explosion

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Table 3 (con't) Convalex 10-Stainless Steel Cell
 Nominal Experimental Conditions
 T_{res} = 650°F T_{cell} = 800°F

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
648	793	72	No Explosion
647	796	145	No Explosion
647	797	270	No Explosion
646	799	420	No Explosion
646	802	510	No Explosion
646	804	616	No Explosion
645	805	755	No Explosion
644	805	655	No Explosion
644	802	550	No Explosion
644	802	460	No Explosion
644	798	384	No Explosion
644	795	240	No Explosion
644	795	165	No Explosion
644	795	70	No Explosion
644	796	112	No Explosion

Table 3 (con't) Convalex 10-Stainless Steel Cell
 Nominal Experimental Conditions
 T_{res} = 650°F T_{cell} = 900°F

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
647	907	72	No Explosion
647	908	105	No Explosion
647	910	303	No Explosion
647	912	412	No Explosion
647	915	540	No Explosion
647	918	703	No Explosion
647	915	584	No Explosion
647	912	480	No Explosion
647	906	372	No Explosion
647	908	270	No Explosion
647	908	195	No Explosion
647	907	107	No Explosion
647	907	78	No Explosion
647	916	586	No Explosion
647	919	715	No Explosion

Table 3 (con't) Convalex 10-Stainless Steel Cell
 Nominal Experimental Conditions

The cell is initially filled to 485 torr of oxygen at 718°F and the temperature of the cell is raised until the vapor pressure exceeds 100 torr

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
718	914	485	No Explosion
761	915	500	No Explosion
801	915	525	No Explosion
846	915	550	No Explosion
880	916	580	No Explosion

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TABLE 4

SPONTANEOUS IGNITION DATA OF DC 704 OBTAINED
IN THE STAINLESS STEEL COMBUSTION CELL

Stoichiometric Mixture: 2.6% DC 704, 97.4% Oxygen

Nominal Experimental Conditions:

$T_{res} = 500^{\circ}\text{F}$, $T_{cell} = 600^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
495	579	45	No Explosion
496	580	60	No Explosion
497	590	255	No Explosion
498	587	125	No Explosion
498	588	505	No Explosion
498	589	635	No Explosion
499	590	760	No Explosion
500	589	593	No Explosion
501	587	455	No Explosion
501	586	395	No Explosion
502	584	300	No Explosion
502	582	250	No Explosion
502	579	150	No Explosion
503	578	50	No Explosion

Table 4 (con't) DC 704-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 530^{\circ}\text{F}$, $T_{cell} = 750^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
527	738	150	No Explosion
529	739	300	No Explosion
530	742	377	No Explosion
532	744	455	No Explosion
532	744	530	No Explosion
533	746	606	No Explosion
535	745	555	No Explosion
535	744	455	No Explosion
535	741	352	No Explosion
537	739	150	No Explosion
537	737	47	No Explosion
537	747	607	No Explosion

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Table 4 (con't) DC 704-Stainless Steel Cell
Nominal Experimental Conditions
 $T_{res} = 590^{\circ}\text{F}$ $T_{cell} = 880^{\circ}\text{F}$

Reservoir Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
495	878	150	No Explosion
496	880	352	No Explosion
496	883	427	No Explosion
497	888	633	No Explosion
498	886	608	No Explosion
499	883	505	No Explosion
499	881	408	No Explosion
499	878	300	No Explosion
500	876	150	No Explosion
500	877	199	No Explosion
501	877	250	No Explosion
502	886	657	No Explosion

Table 4 (con't) DC 704-Stainless Steel Cell
Nominal Experimental Conditions
 $T_{res} = 650^{\circ}\text{F}$ $T_{cell} = 900^{\circ}\text{F}$

Reservoir Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
496	880	300	No Explosion
496	881	372	No Explosion
455	883	497	No Explosion
523	885	734	No Explosion
580	887	580	No Explosion
624	887	607	No Explosion
662	888	584	No Explosion
703	889	657	No Explosion
746	889	683	No Explosion
766	890	707	No Explosion

Table 4 (con't) DC 704-Stainless Steel Cell
Nominal Experimental Conditions
 $T_{res} = 590^{\circ}\text{F}$ $T_{cell} = 600^{\circ}\text{F}$

Reservoir Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
516	607	222	No Explosion
513	608	250	No Explosion
512	608	606	No Explosion
510	609	250	No Explosion
508	609	352	No Explosion
508	609	459	No Explosion
507	609	557	No Explosion
506	621	708	Explosion

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Table 4 (con't) DC 704-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 500^{\circ}F$ $T_{cell} = 710^{\circ}F$

Reservoir Temp. ($^{\circ}F$)	Combustion Cell Temp. ($^{\circ}F$)	Total Pressure (torr)	Comments
502	710	352	No Explosion
502	711	455	No Explosion
502	720	557	S. P. I. S. T. I.
503	721	607	No Explosion
505	721	657	No Explosion
509	709	300	
509	710	557	No Explosion
509	710	403	S. T. I.
510	719	505	S. P. I. S. T. I.
510	720	607	No Explosion
510	720	657	No Explosion

Table 4 (con't) DC 704-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 510^{\circ}F$ $T_{cell} = 800^{\circ}F$

Reservoir Temp. ($^{\circ}F$)	Combustion Cell Temp. ($^{\circ}F$)	Total Pressure (torr)	Comments
513	803	760	S. T. I.
513	806	352	No Explosion
513	806	455	Explosion
512	805	557	No Explosion
512	805	633	Explosion
511	805	300	S. T. I.
511	808	377	S. T. I.
511	818	505	No Explosion
510	806	760	S. T. I.
510	813	325	S. T. I.
510	806	148	No Explosion
510	807	197	S. T. I. S. P. I.
509	813	403	No Explosion
509	816	455	No Explosion

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Table 4 (con't) DC 704-Stainless Steel Cell
 Nominal Experimental Conditions
 $T_{res} = 500^{\circ}\text{F}$ $T_{cell} = 900^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
502	893	300	No Explosion
502	900	325	No Explosion
502	893	97	S. T. I.
502	899	225	S. T. I.
502	893	47	No Explosion
502	893	148	S. T. I.
502	899	403	S. T. I.
502	903	556	S. T. I.
502	893	97	No Explosion
502	894	198	S. T. I.
502	901	428	No Explosion
502	893	97	S. T. I.
502	901	352	No Explosion

Table 4 (con't) DC 704-Stainless Steel
 Nominal Experimental Conditions

The cell is initially filled to 40 torr of oxygen at 510°F and the temperature of the cell is raised until the vapor pressure exceeds 100 torr

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
516	893	300	Explosion
514	902	400	No Explosion
512	896	100	Explosion
512	899	300	No Explosion
513	900	410	No Explosion
513	897	40	Explosion
513	898	190	No Explosion

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TABLE 5
SPONTANEOUS IGNITION DATA OF T. C. P. OBTAINED IN THE
STAINLESS STEEL COMBUSTION CELL

Stoichiometric Mixture: 3.8% T. C. P.; 96.2% Oxygen

Nominal Experimental Conditions:

$T_{res} = 520^{\circ}\text{F}$, $T_{cell} = 620^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
521	603	10.7	No Explosion
521	606	48.7	No Explosion
521	609	61.4	No Explosion
522	612	107	No Explosion
522	613	120	No Explosion
522	615	155	No Explosion
523	615	163	No Explosion
523	617	201	No Explosion
523	619	252	No Explosion
523	620	290	No Explosion
523	624	322	No Explosion
523	625	358	No Explosion
523	628	399	No Explosion
523	629	429	No Explosion
524	631	467	No Explosion
524	631	506	No Explosion
524	632	531	No Explosion
524	632	562	No Explosion
524	633	595	No Explosion
525	633	620	No Explosion
525	635	651	No Explosion
525	635	671	No Explosion
525	636	705	No Explosion
525	636	735	No Explosion

Table 5 (con't) TGP-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 530^{\circ}\text{F}$, $T_{cell} = 700^{\circ}\text{F}$

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
528	702	10.7	No Explosion
528	704	112.0	No Explosion
529	707	252	No Explosion
529	710	379	No Explosion
530	712	506	No Explosion
531	714	620	No Explosion
532	716	735	No Explosion

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Table 5 (con't) TCP-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 500^{\circ}\text{F}$ $T_{cell} = 830^{\circ}\text{F}$

Reservoir Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
507	827	79.1	No Explosion
507	827	171	No Explosion
507	829	247	No Explosion
507	831	374	No Explosion
507	834	500	No Explosion
507	837	652	No Explosion
507	834	506	No Explosion
507	831	378	No Explosion
507	827	252	No Explosion
507	827	241	No Explosion
507	925	32	No Explosion
507	837	506	No Explosion
507	925		

Table 5 (con't) TCP-Stainless Steel Cell

Nominal Experimental Conditions

$T_{res} = 525^{\circ}\text{F}$ $T_{cell} = 900^{\circ}\text{F}$

Reservoir Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
513	913	608	No Explosion
518	905	292	No Explosion
521	901	145	No Explosion
525	901	48.3	No Explosion
5.26	899	0	No Explosion
5.26	900	12.7	No Explosion
5.26	905	125	No Explosion
5.26	902	247	No Explosion
5.26	903	317	No Explosion
5.26	905	378	No Explosion
5.26	907	449	No Explosion
5.26	909	550	No Explosion
5.26	911	650	No Explosion
5.26	901	0	No Explosion

Table 5 (con't) T.C.P.-Stainless Steel Cell

Nominal Experimental Conditions

The cell is initially filled to 373 torr of oxygen at 514°F and the temperature of the cell is raised until the vapor pressure exceeds 100 torr

Reservoir Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
514	899	373	No Explosion
541	905	396	No Explosion
585	907	410	No Explosion
628	908	412	No Explosion
660	908	414	No Explosion
707	909	523	No Explosion
748	912	530	No Explosion
757	914	560	No Explosion
764	915	605	No Explosion

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Table 5 (con't) TCP-Stainless Steel Cell

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
770	917	632	No Explosion
780	921	683	" "
790	921	708	" "
801	924	740	" "
818	926	668	" "
835	929	582	" "
850	934	453	" "
892	979	378	" "

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TABLE 6

**SPONTANEOUS IGNITION DATA OF CONVOIL 20 OBTAINED IN THE
STAINLESS STEEL COMBUSTION CELL**

Stoichiometric Mixture: 2.4% Convoil 20; 97.6% Oxygen

Nominal Experiment Conditions:

$T_{res} = 500^{\circ}\text{F}$, $T_{cell} = 610^{\circ}\text{F}$

Res. Temp. ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (Torr)	Comments
486	609	45	No Explosion
488	611	270	No Explosion
488	613	415	No Explosion
489	615	555	No Explosion
489	617	760	No Explosion
490	617	734	No Explosion
490	615	605	No Explosion
490	612	480	No Explosion
490	610	430	No Explosion
490	608	250	No Explosion
490	606	197	No Explosion
490	606	125	No Explosion
490	605	70	No Explosion
489	605	45	No Explosion

Table 6 (con't) Convoil 20- Stainless Steel

Nominal Experiment Conditions

$T_{res} = 540^{\circ}\text{F}$ $T_{cell} = 700^{\circ}\text{F}$

Reservoir Tempa ($^{\circ}\text{F}$)	Combustion Cell Temp. ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
534	707	250	Explosion
535	708	428	Explosion
535	709	530	
541	706	250	No Explosion
542	708	403	Explosion
542	710	634	No Explosion

Table 6 (con't) Convoil 20-Stainless Steel Cell

Nominal Experiment Conditions

$T_{res} = 550^{\circ}\text{F}$ $T_{cell} = 800^{\circ}\text{F}$

Reservoir Tempa ($^{\circ}\text{F}$)	Combustion Cell Temp ($^{\circ}\text{F}$)	Total Pressure (torr)	Comments
554	802	325	No Explosion
556	803	378	No Explosion
557	802	479	S. P. I.
558	804	580	No Explosion
541	805	555	No Explosion
538	802	300	S. P. I. & S. T. I.

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Table 6 (con't) Convoil 20-Stainless Steel Cell
Nominal Experiment Conditions

The cell is initially filled to 98 torr of oxygen
at 511°F and the temperature of the cell is raised
until the vapor pressure exceeds 100 torr

Reservoir Temp. (°F)	Combustion Cell Temp. (°F)	Total Pressure (torr)	Comments
511	802	403	No Explosion
511	802	455	No Explosion
511	803	505	No Explosion
511	804	555	No Explosion
511	804	607	No Explosion
510	805	657	No Explosion
510	806	708	No Explosion
510	796	98	No Explosion
510	796	198	No Explosion
510	799	300	No Explosion
511	800	403	No Explosion
511	802	505	No Explosion
511	804	605	No Explosion
512	799	403	No Explosion
512	800	455	No Explosion
512	802	530	No Explosion
512	802	607	Explosion
512	796	148	Explosion
513	795	352	No Explosion
513	800	455	No Explosion
514	795	275	No Explosion
514	798	453	No Explosion
514	800	505	No Explosion
514	794	250	No Explosion
514	796	148	S. T. I.
515	797	250	No Explosion

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APPENDIX B

APPARATUS AND PROCEDURES

In the course of this experiment, several apparatus were constructed and used. This equipment and the procedures associated with their operation are described in this appendix.

In the combustion phase of this project, two glass systems and a stainless steel system have been utilized. In addition, ten glass combustion cells were constructed for the two week catalytic experiment. Detonation apparatus were constructed to evaluate the possibility of liquid phase detonation. A spark ignition system was constructed which could be used for all the spark tests and its design is considered in some detail in Appendix D. In the high vacuum phase, a thirty-five inch and a six inch diffusion pump were used in the test program. Techniques for measuring backstreaming rates were also developed.

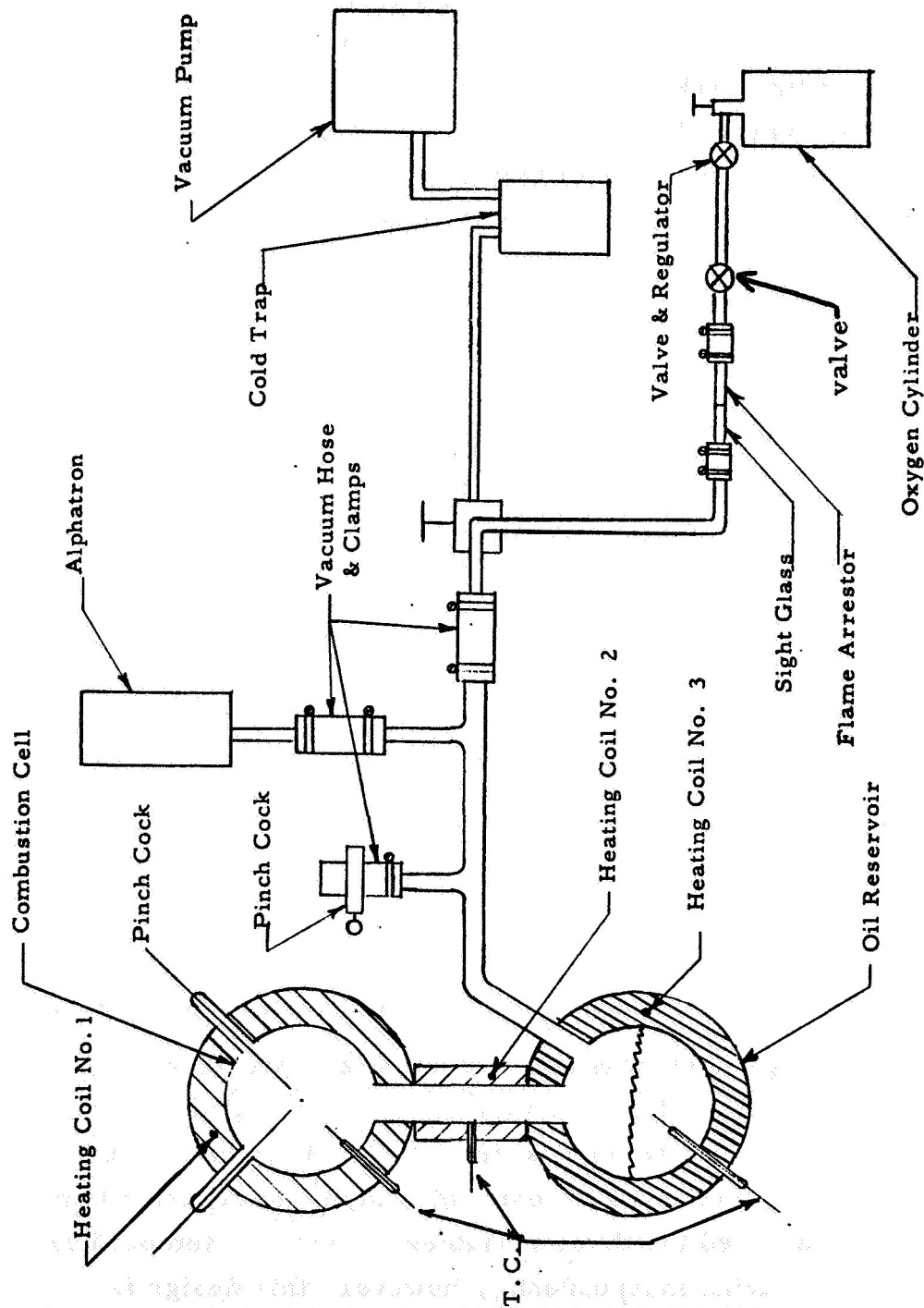
A Combustion Phase

1. Glass System

A major problem existed in the design of a closed bomb combustion apparatus because the oils being investigated are condensable at room temperatures. The pressure gage of the cell must be at room temperature causing oil to condense in it. This condensation changes the oil-oxygen concentration present in the cell. In order to circumvent this problem the pressure gage should be at the same temperature as the rest of the system. Manufacturers of high vacuum gages were consulted and it was found that none had, or could make a gage which could withstand 1000° F, the maximum operating temperature. Initially, a system was set up with an Alphatron pressure gage at ambient temperature with the hope that the oil would not diffuse rapidly into the gage. Preliminary experiments indicated that the amount of condensation was significant and thus another solution was sought.

The solution to this problem was attained and one of the apparatus with this solution incorporated in it is shown in Fig. 1. The combustion cell is the top bulb, the bottom bulb is an oil reservoir. The heaters on these bulbs are independent so that the cell and reservoir may be at different temperatures. This design allowed the three variables, pressure, temperature, and oil-oxygen ratio, to be varied independently, however, this design is no

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* T.C. = Thermocouple

Fig. 1 GLASS COMBUSTION APPARATUS

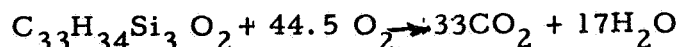
longer, strictly a closed combustion bomb. The temperature of the reservoir may be adjusted to correspond to a desired vapor pressure. Since the connection between the cell and reservoir is open, the vapor pressure will represent the partial pressure of the oil in the cell. Oxygen admitted from the oxygen tank may be used to supply the desired partial pressure of oxygen. The cell temperature may be varied to any temperature above that of the oil reservoir. The heater on the glass tube between the cell and reservoir is used to keep the temperature of this section between that of the two bulbs so that no oil will condense in this section and so it will not provide the ignition source.

Since the pressure gage is connected to the reservoir and not to the cell, any oil condensing in it will be from the reservoir and not the cell. Since pressures throughout the system should be very close to uniform, this gage measures the pressure in the cell.

It was necessary to have thermocouple wire and spark wire leads extend through the glass. This required a special sealing technique. Thermosetting glass tape was soaked in a solvent such as benzene to remove the adhesive and then dried. The remaining cloth was dipped into a thin solution of Saureisen cement (cement plus solvent) and the cloth wrapped around the glass blown connection. After this dried, another layer was added. The temperature which this seal withstood was adequate to allow a cell temperature of 900°F.

This combustion apparatus was used to investigate very thoroughly the explosive properties of the oil selected as the best oil to use, DC-750. The procedure followed for conducting the explosion experiments was the following:

The system was evacuated, purged with oxygen and re-evacuated. The oil reservoir was heated to a temperature T_1 corresponding to the desired vapor pressure of the oil. The lowest oil pressure investigated corresponded to the partial pressure of oil necessary to give a stoichiometric mixture of oxygen and oil at the desired minimum total pressure. This pressure was the lowest total pressure considered. The stoichiometric ratio was determined from the reaction;



which means that the total stoichiometric pressure, P_T , corresponding to a vapor pressure of oil of P_{oil} is given by;

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$$P_T = 45.5 \times P_{oil}$$

The temperature of the cell was set at a temperature above that of the reservoir and then the oxygen was admitted to the cell. At various pressure levels, ignition of the gas mixture was attempted with a spark.* The cell temperature and pressure were monitored at all times for an indication of explosion. After the maximum pressure was reached, the cell was re-evacuated and purged and the temperature of the cell was raised, and the same procedure repeated. The maximum temperature investigated was 900°F. Although the temperature of the oil is much higher than that which can be expected in a diffusion pump, the purpose of this procedure is to investigate the hazard potential under more arduous conditions than encountered in a diffusion pump so that a safety factor will be inherent in the results obtained. After all these temperatures were investigated, the temperature of the reservoir was raised to give a higher vapor pressure and the above procedure was repeated. The entire procedure was repeated until the maximum vapor pressure investigated corresponded to the maximum total stoichiometric pressure of interest (about 400 torr).

The glass combustion system was the last one which was constructed and it incorporated all of the properties which we had found to be lacking in the former combustion cells. If any future work is to be done of a similar nature, this is the equipment or design which should be used.

2. Stainless Steel Apparatus

The original stainless steel apparatus was modified to include the characteristics inherent in the glass system. The diagram of this equipment is shown in Fig. 2. This apparatus could not be used for experiments below one millimeter in pressure because of leaks through the valve stems even though all metal to metal contacts were silver soldered to prevent leaks.

* The spark system is described later in this appendix.

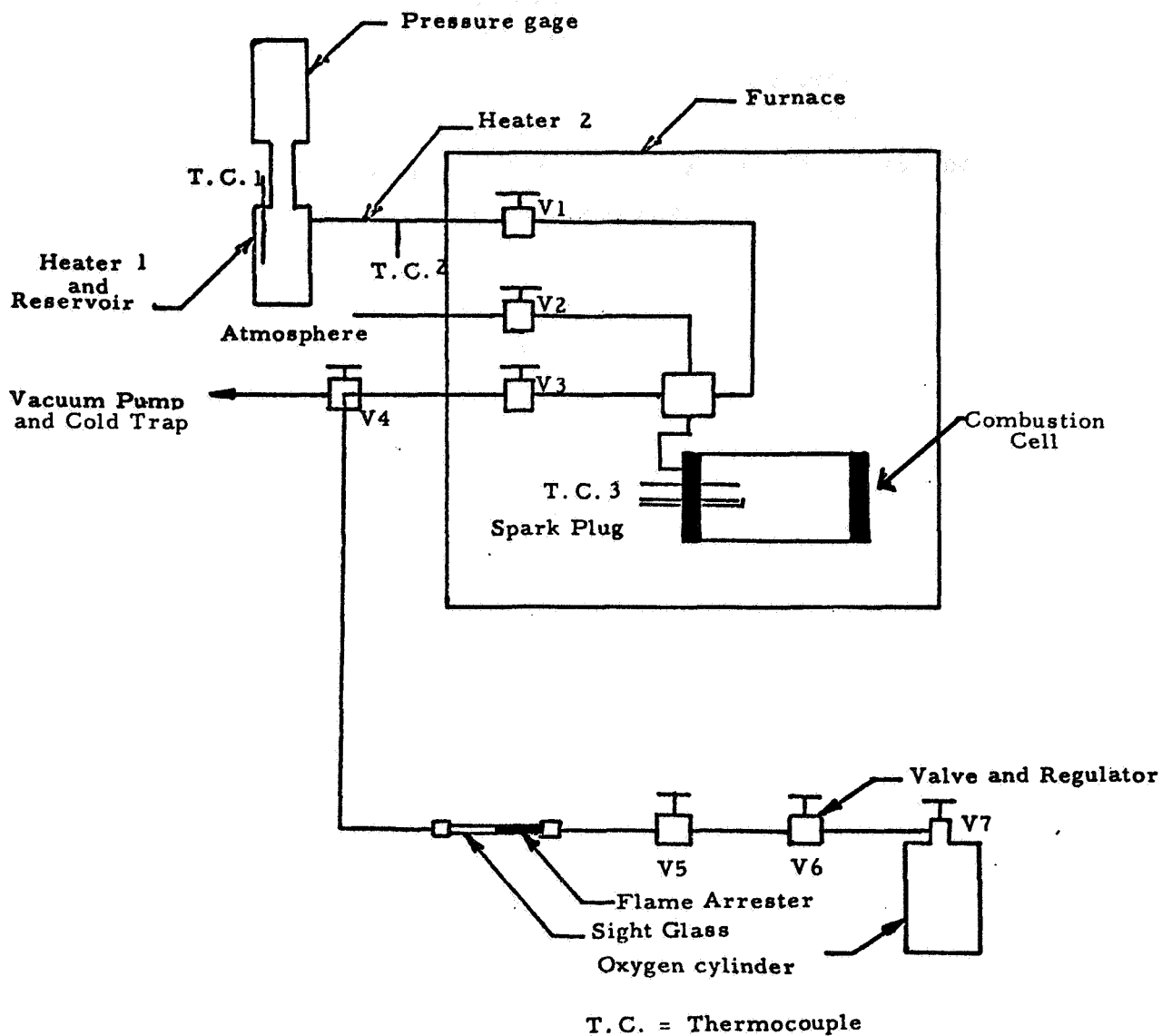


Fig. 2. STAINLESS STEEL COMBUSTION APPARATUS (REVISED)

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A series of tests was conducted in the stainless steel apparatus for each of the five oils under consideration. The procedure followed was initiated by adjusting the vapor pressure of oil in the reservoir to about 10 torr. A 700° F temperature in the cell was selected and pure oxygen was injected in varying amounts until atmospheric pressure was attained. The cell pressure and temperature were monitored at all times for an indication of explosion. The same procedure was repeated at 800° F and 900° F and at 600° F if the temperature of the reservoir was below 600° F. The final test for each oil was for very oil rich conditions. Oxygen was admitted to a pressure of 300 torr with the cell temperature at 900° F and then the temperature of the oil was raised to about 800° F. This increased the vapor pressure of the oil to over 100 torr. Again the purpose of this procedure was to investigate the hazard potential under more arduous conditions than encountered in a diffusion pump so that a safety factor would be inherent in the results obtained.

The detailed procedure of the first combustion test is the following:

- 1) Close all valves (V1, V2, V3, V4, V5, V6, V7).
- 2) Heat furnace to temperature t_3 .
Heat heater 1 to temperature t_1 .
Heat heater 2 to temperature t_2 .
- 3) Open V4 and evacuate this line.
- 4) Open V5 and V6 and open the regulator all the way.
- 5) Close V5, close regulator, open V7, close V7 and, then, V6.
Set regulator at 5 psig.
- 6) Open V3 to evacuate cell.
- 7) Open V1 slowly to evacuate reservoir. Close V1 immediately when pressure reaches a minimum.
- 8) Close V3, open V1, then, wait long enough for the vapor from the liquid to propagate into the cell.
- 9) Close V4, and open V5.
- 10) Slowly open V3 until the desired pressure is reached in the cell. Wait for equilibrium of the gas temperature and pressure P_2 .

Continue this process until the maximum test pressure is reached.

11) Close V5, open V4 slowly to evacuate the line.

12) Evacuate the cell at even pressure intervals with V3.

This procedure was repeated for all desired temperatures. For the last test, this procedure was repeated to step 10 with the exception of selecting P_1 to be about 300 torr. Then the temperature of the reservoir was raised until an explosion occurred or until a vapor pressure of 100 torr was reached.

3. Initial Glass System

A diagram of this system is shown in Fig. 3. It was used simply to begin the experiments while the stainless steel cell was being constructed. It has several disadvantages and its diagram is shown only for the sake of completeness.

4. Catalytic Equipment

A picture of the combustion cells used in the catalytic experiments is shown in the Fig. 4 and a diagram of a single cell is shown in Fig. 5. The single cell consists of the main combustion cell which has oxygen, oil, and shavings of one of the metal catalysts in it. A thermocouple is attached to the outside wall of this bulb. A heater surrounds the entire cell. A manometer is used to measure pressure. The height of the mercury is measured by the resistance of a nichrome wire in the right leg. Electrodes were also installed in the bomb so that a spark could be dissipated in the cell. The pressure and temperature of each of the cells could be recorded once every 25 seconds by use of the digital multiplexing voltmeter shown in the right of Fig. 4.

The manifold shown in this figure was used to charge the cells. Initially, the entire system was evacuated by use of a vacuum pump with a cold trap (not shown) and purged and re-evacuated. Variacs were used to adjust the temperature of each of the cells to approximately 450° F. Three of the heaters were connected to one variac so that each of these heaters could not be adjusted independently. After the temperatures were adjusted to approximately 450° F, about 400 torr of oxygen was added to the cells.

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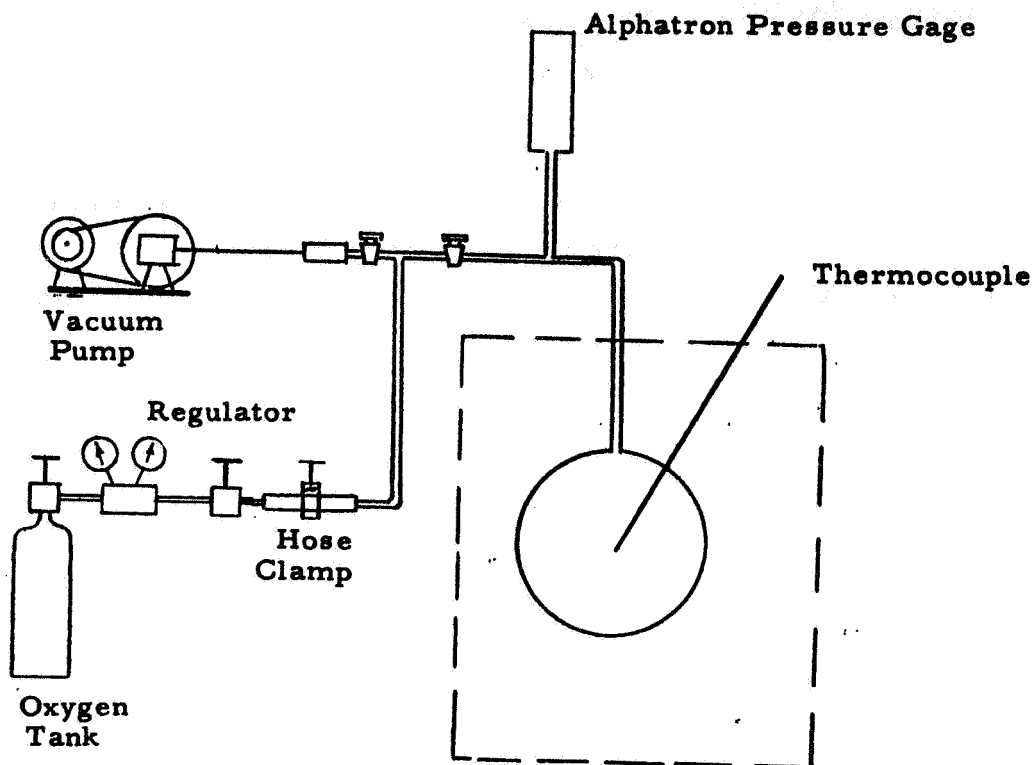


Fig. 3 GLASS COMBUSTION SYSTEM

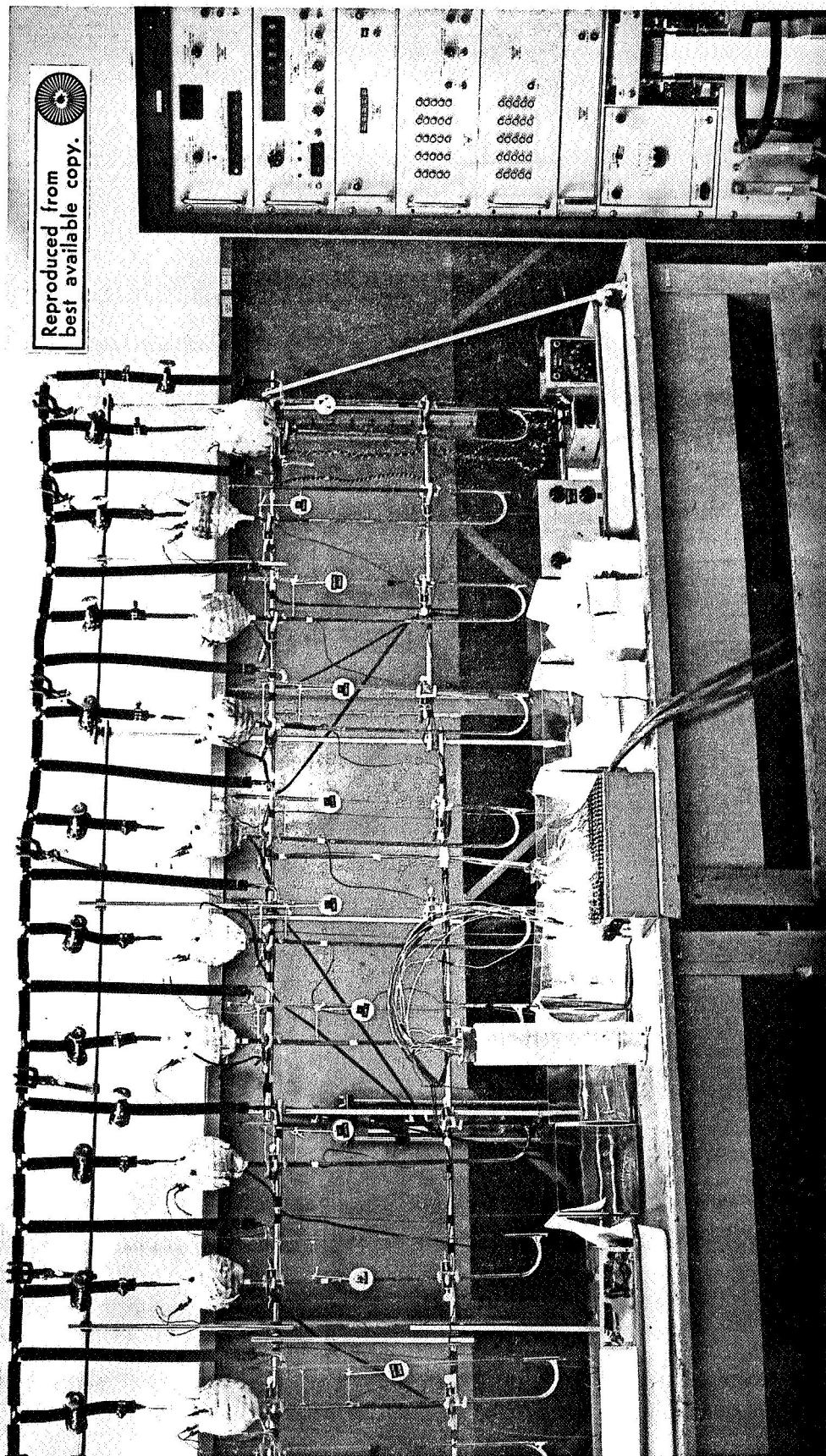


Fig. 4 EXPERIMENTAL APPARATUS USED FOR THE CATALYTIC EXPERIMENTS

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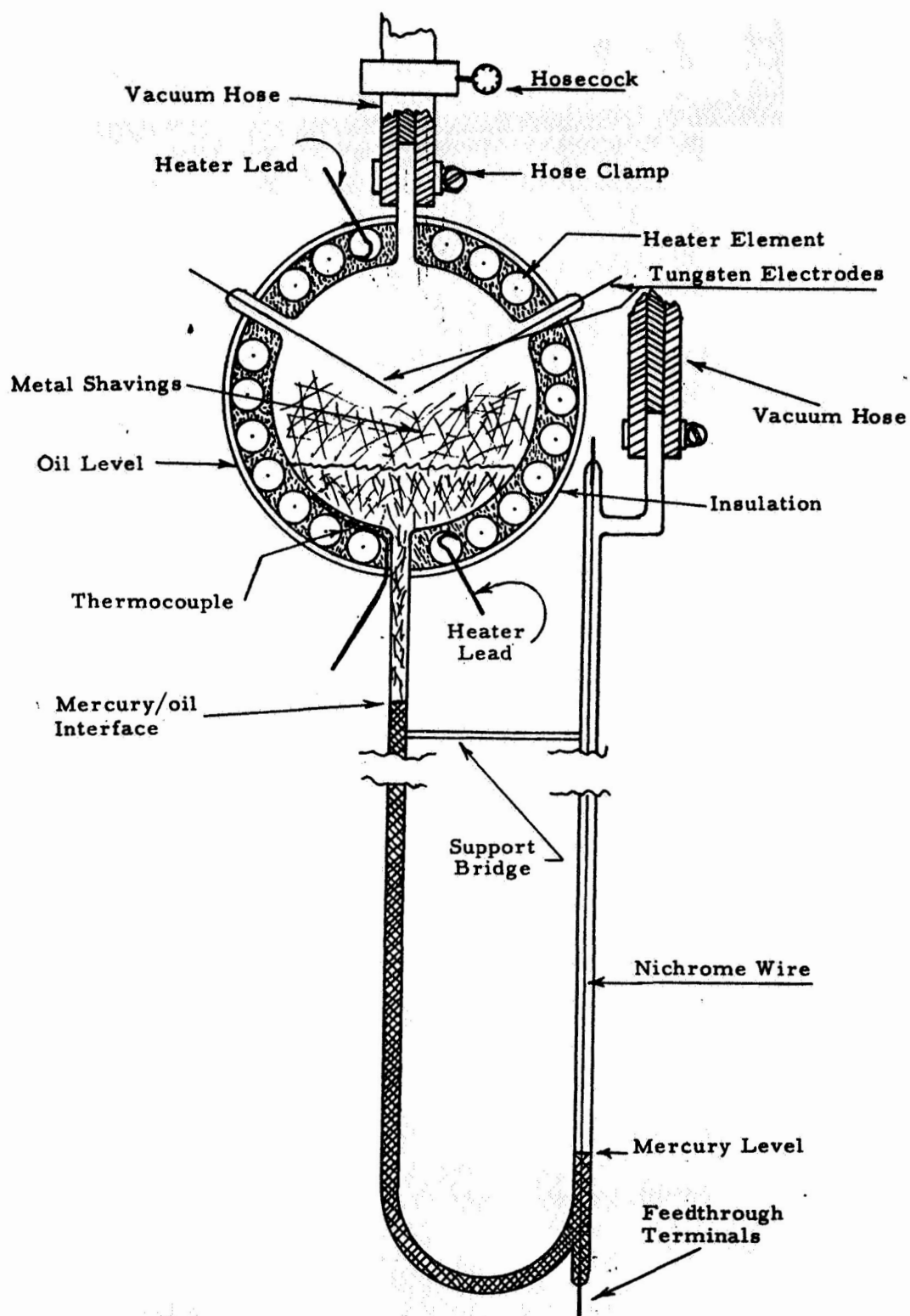


Fig. 5 A CATALYTIC COMBUSTION CELL
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Then, each of the cells was closed off with a hose clamp and the manifold was opened to the atmosphere. Once started, the tests were run for two weeks and pressures were recorded continuously. The pile of paper on the table in Fig. 4 represents the data obtained in five days of operation. As a point of interest, the paper upon which the data of the two week run was recorded, occupies a volume of approximately 2.5 cu ft.

5. Detonation Apparatus

Detonation was attempted on eight different samples of oil. The detonation equipment used for each of these samples is shown in Fig. 6.

The blasting cap detonated the tetryl booster. If the booster initiates a detonation in the oil, severe scars are formed by metal flow at the impingement surface of the detonation wave. If the oil has not detonated, the witness block will remain unmarked. The method of determining detonation has been widely and very successfully used throughout the world in the explosives field.

The pressed tetryl pellets used as the booster in our set up are the standard type used in this kind of testing with all secondary explosives. Pressed tetryl detonates with a reaction velocity in excess of 20,000 fps, and surface pressures in the order of 4,000,000 psi.

The boosters subject the oils to conditions far in excess of several orders of magnitude of severity as compared to those to which these oils are ordinarily subjected in diffusion pumping systems.

6. Spark Ignition System

The diagram of the spark circuit is shown in Fig. 7. The background and design of this system is described in Appendix D.

To operate this system, the lead wires are attached to the spark electrodes. (Fig. 7 shows these leads connected to a spark plug.) The value of capacitance may be varied in increments of 25 μ fd from 25 to 825 μ fd. The voltage output from the high voltage supply is controlled by the variac shown and is variable between 0 to 10,000 volts DC. A certain energy build up in the capacitors may be obtained by selecting a value of capacitance and voltage. When this energy has been built up, the switch is thrown and the energy is dissipated across the spark gap. The time of energy build up may be varied by using resistors of different

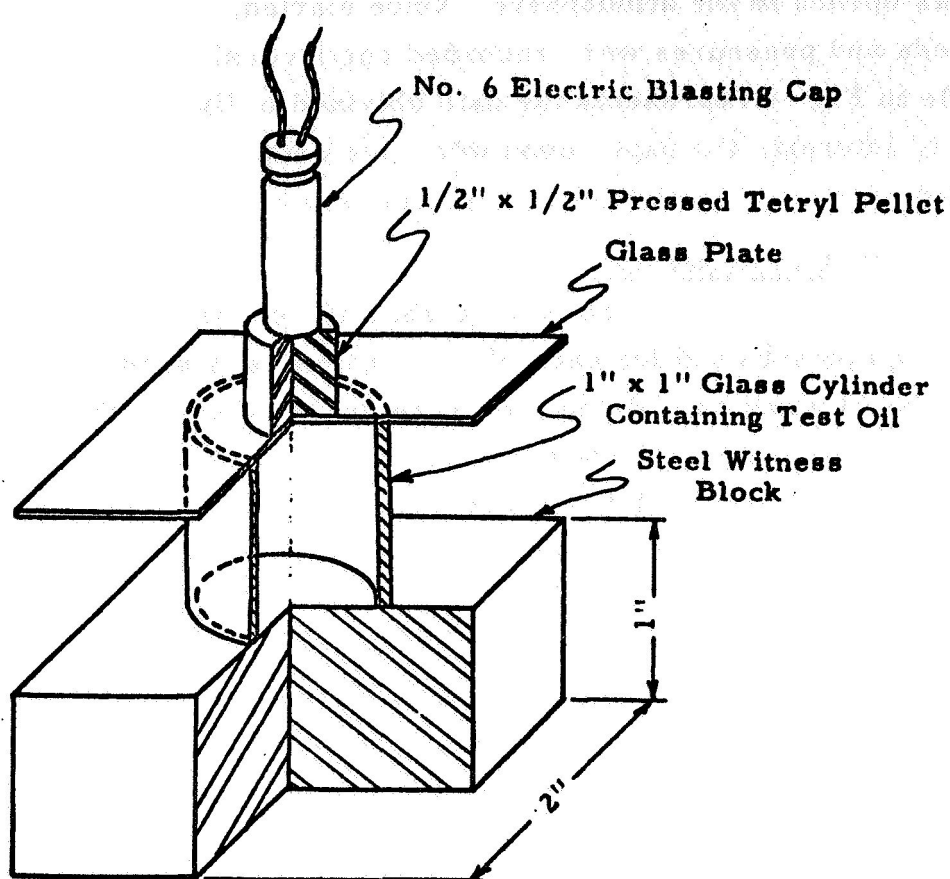


Fig. 6 EXPERIMENTAL SET-UP FOR DETERMINATION
OF CONDENSED PHASE DETONATION IN LIQUIDS

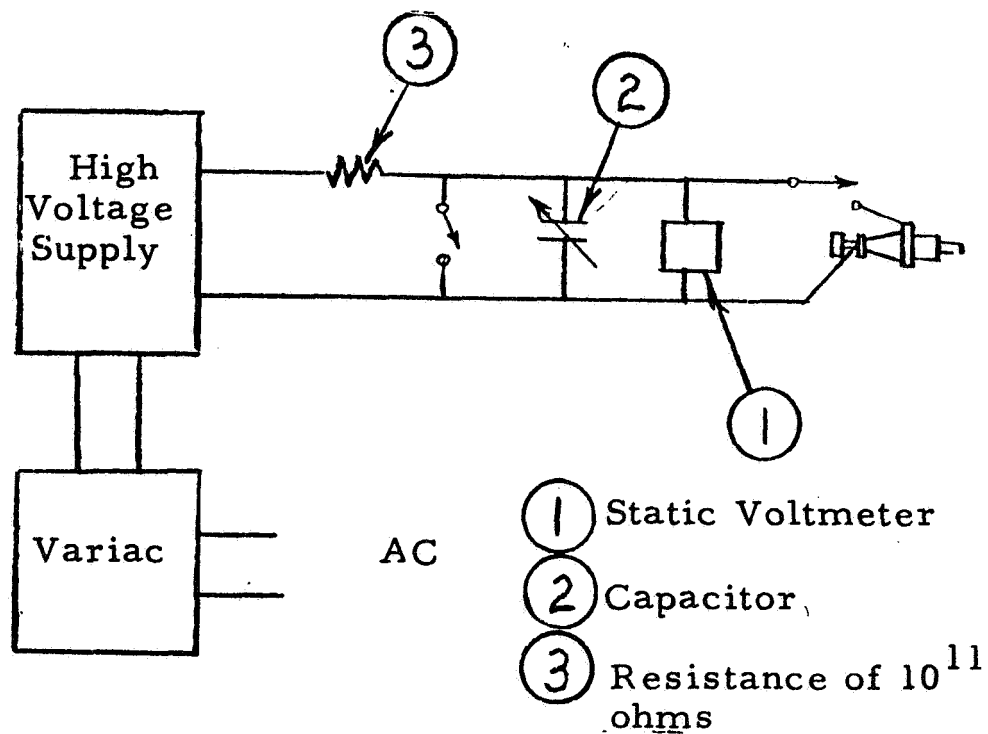


Fig. 7 DETAILED DIAGRAM OF SPARK CIRCUIT

resistance. Two resistors were made up; one with a resistance of 10^9 ohms the other with a resistance of 10^{11} ohms. It was necessary to use resistors of this magnitude so that the switch could be disengaged after one spark occurred and before the second began to build up.

B. High Vacuum Phase

1. Vacuum Equipment

The vacuum equipment assembled for this program consists of two high vacuum stations, one pumped by a 6 inch, 15,000 l/sec. diffusion pump and one by a 35 inch, 50,000 l/sec. diffusion pump. The equipment is shown schematically in Fig. 8. Two 110 cfm mechanical vacuum pumps are coupled in parallel with valving to permit either or both pumps to back either diffusion pump*. The diffusion pumps were fitted with water cooled, chevron type baffles. No high vacuum valves or separate rough pumping equipment was utilized. Initially, a single 36 inch dia. by 48 inch high stainless steel bell jar was incorporated with a common overhead hoist to permit utilization of the bell on either pump station. However, scheduling of tests required simultaneous operation of both stations and an 18 inch dia. by 18 inch high bell jar was fitted to the 6 inch pump. The 36 inch bell was used solely with the larger pump.

Instrumentation included a multi-station Pirani type gage to monitor foreline pressures and hot-cathode ionization gauges to measure chamber pressures. Thermocouples were installed in the 35 inch pump to monitor boiler temperature, fluid temperature at the middle jet and baffle temperature. Fail-safe circuitry was incorporated to prevent damage to the equipment in the event of loss of electrical power, cooling water, or foreline vacuum.

Both bell jars were fitted with inlet valves which were used for admission of oxygen during the test and for venting the system to atmosphere after the test. The 6 inch system was also fitted with three automotive type spark plugs; one in the bell jar, one in the diffusion pump barrel and one in the foreline.

Auxillary equipment included a helium leak detector for assuring leak-tight operation, an automotive type spark coil for sparking the spark plugs, and an oxygen admission system. The oxygen admission system consisted of a gaseous oxygen storage bottle with pressure regulator, a

* Equipment is listed in Table 1.

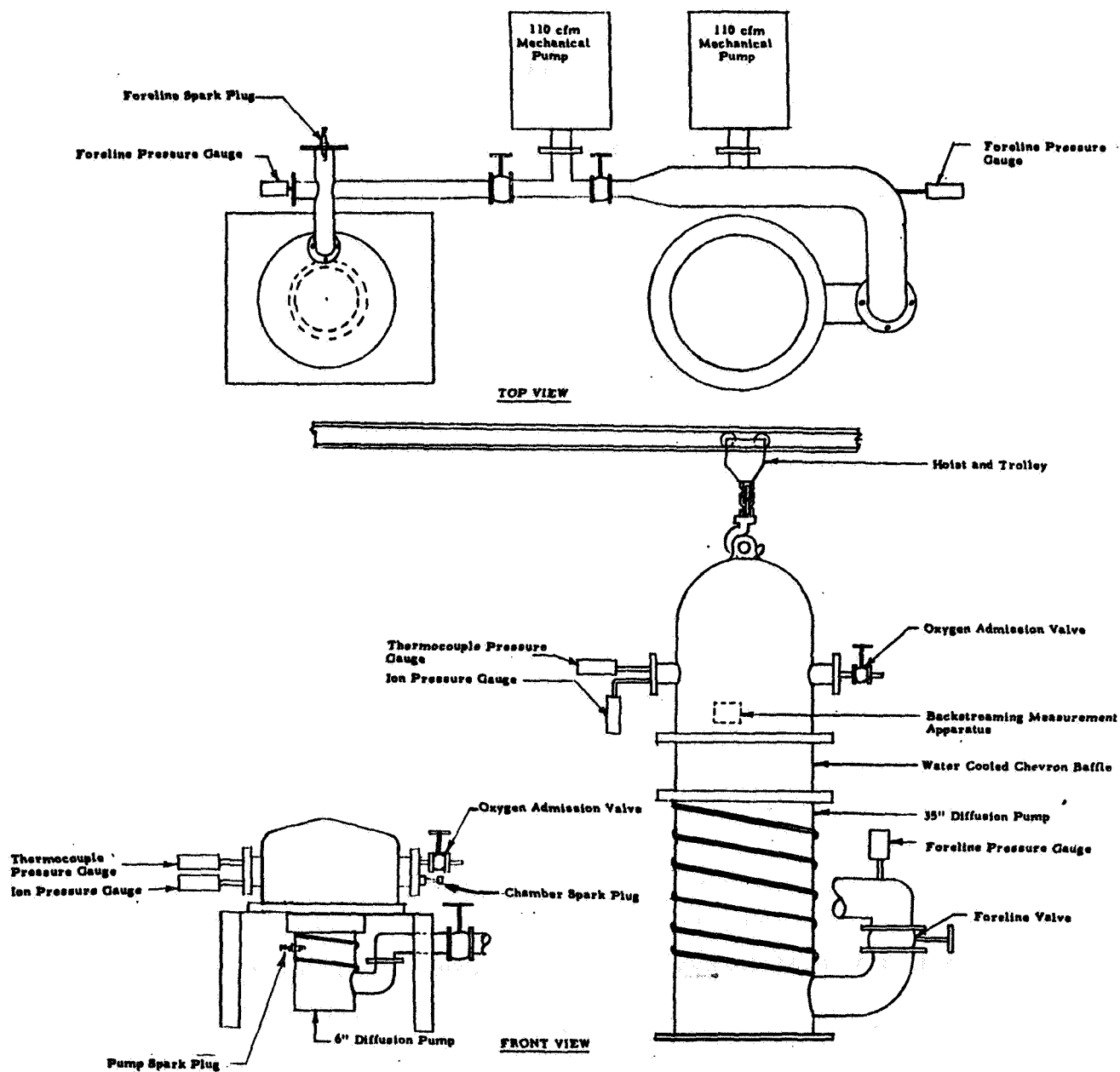


Fig. 8

HIGH VACUUM APPARATUS

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TABLE 1
VACUUM AND ACCESSORY EQUIPMENT

Equipment	Supplier	Model
6" Diffusion Pump	NRC Equipment Corp.,	Model HS 6-1500
35" Diffusion Pump	CVC Corp.,	Model PMC 50,000
6" Baffle	CVC Corp.,	Model BC 61
35" Baffle	CVC Corp.,	Model BC 350
Mechanical Backing Pumps	Kinney Div.	Model 8-8-10
Foreline Pressure Gage	CVC Corp.,	Model GMA-140
6" Chamber Pressure Gage	Control Unit Magnevac Gage Tube Type	Model 426 VGIA
35" Chamber Pressure Gage	Control Unit Varian Asso., Gage Tube Type	Model 971-003 VGIA
Spark Plugs	Champion Gap = 0.10	Model D89D

75 cubic foot polyethylene bag oxygen reservoir, an in-line pressure gage and valve for regulating the oxygen flow, and a small mechanical pump for evacuating the oxygen inlet line prior to test. The oxygen inlet lines were 3/8 inch bore, thick wall Tygon hose for the 6 inch pump station, and 1 1/2 inch iron pipe for the 35 inch pump station. The oxygen admission system is shown schematically in Fig.9 . The admission system was located outside the building housing the vacuum equipment after Test 2-1.

2. Backstreaming Test Equipment

Since an ellipsometer was not readily available, a fixed angle, polarizing spectroscope was assembled from components on hand. The apparatus is shown in Fig. 10. In operation, a monochromatic, collimated linearly polarized light was reflected from the specimen in 45° azimuth with the angle of incidence set at 75°. The reflected beam was restored to linearity by a phase compensator and the azimuth of the restored linear polarization was measured with the analyzer. A double-null measurement was made on each specimen plate, before and after exposure. The four readings thus obtained permitted calculation of the optical thickness to physical thickness.

Contact angles were measured with a goniometer assembled from components available in the laboratory. A telescope with 60° cross hairs horizontal specimen table. The angle formed at the interface of a drop of liquid placed on a horizontal specimen could thus be measured with a precision greater than one degree of arc.

Specimens were exposed to oil vapors for short periods during pump operation by placing them in the shutter box so that the exposed slide viewed the water cooled baffle at a position 12 inches from the edge of the pump and at an elevation of 5 inches above the baffle. For longer exposures, specimens were mounted outside the shutter box in a similar position.

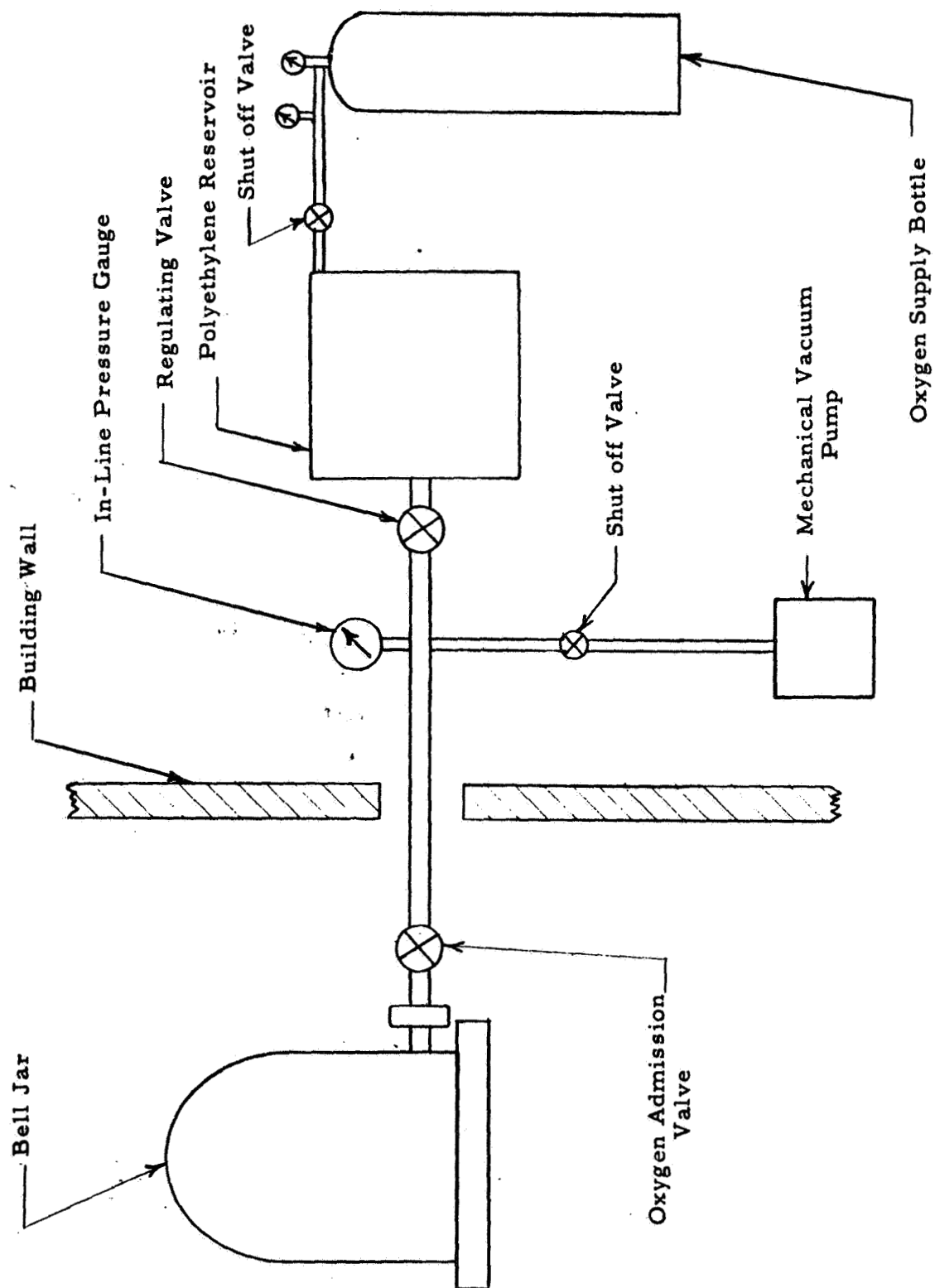


Fig. 9 OXYGEN ADMISSION SYSTEM

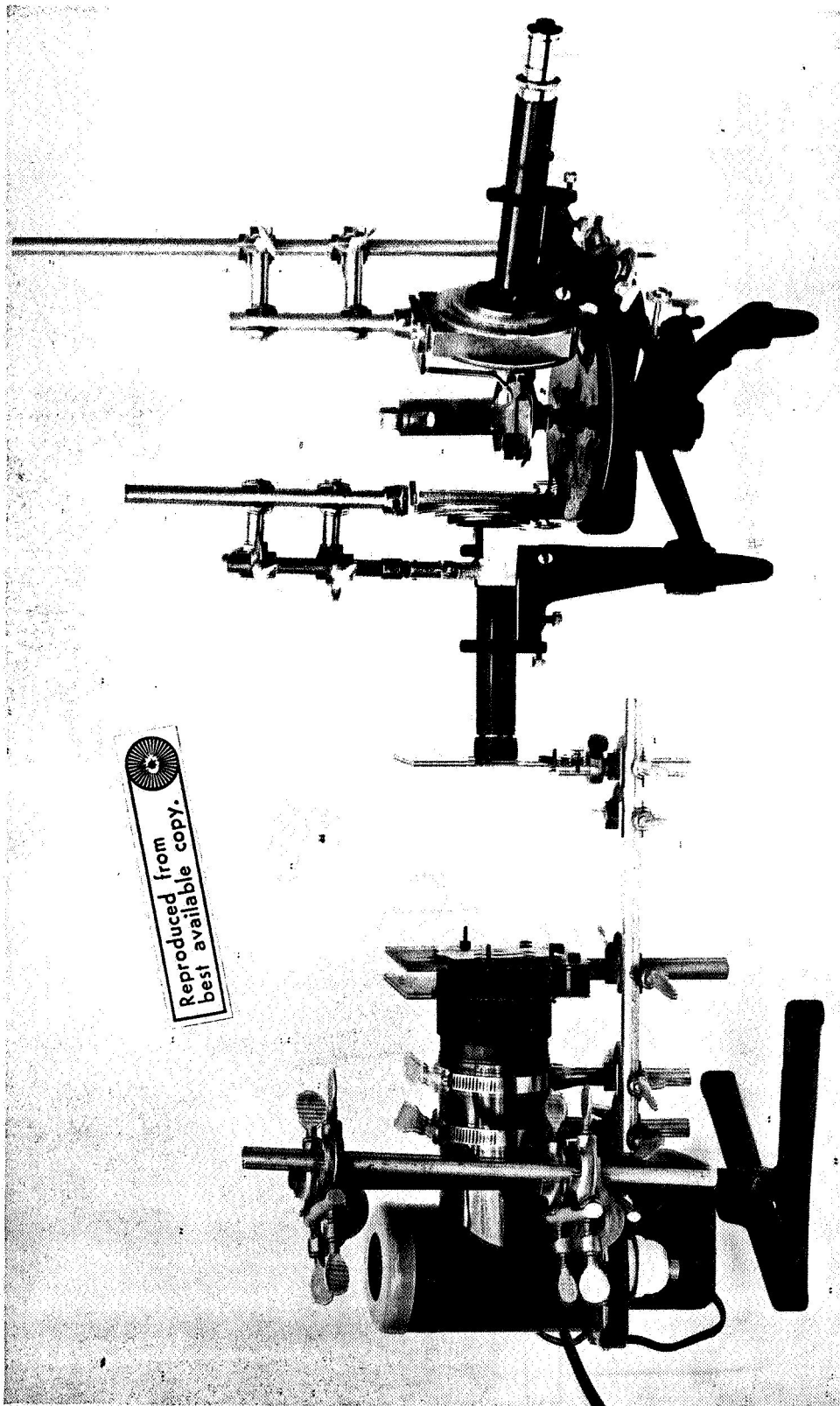


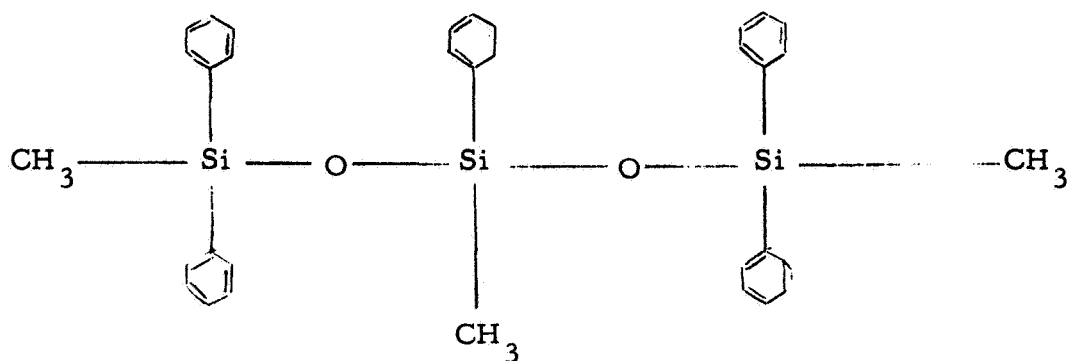
Fig. 10 FIXED ANGLE, POLARIZING SPECTROSCOPE

APPENDIX C

PHYSICAL PROPERTIES OF DIFFUSION PUMP OILS

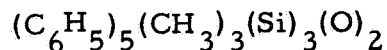
DIFFUSION PUMP OIL: DC-705

MOLECULAR STRUCTURE



 = PHENYL GROUP

FORMULA



MOLECULAR WEIGHT

546.0

TECHNICAL NAME

Pentaphenyltrimethyltrisiloxane

STOICHIOMETRIC FORMULA



SiO_2 is a solid

ISOTHERMAL PRESSURE RATIO

$$\frac{P_2}{P_1} = \frac{50.0}{44.5} = 1.1$$

THERMAL STABILITY

Decomposition begins to occur at 700° F (isoteniscope)

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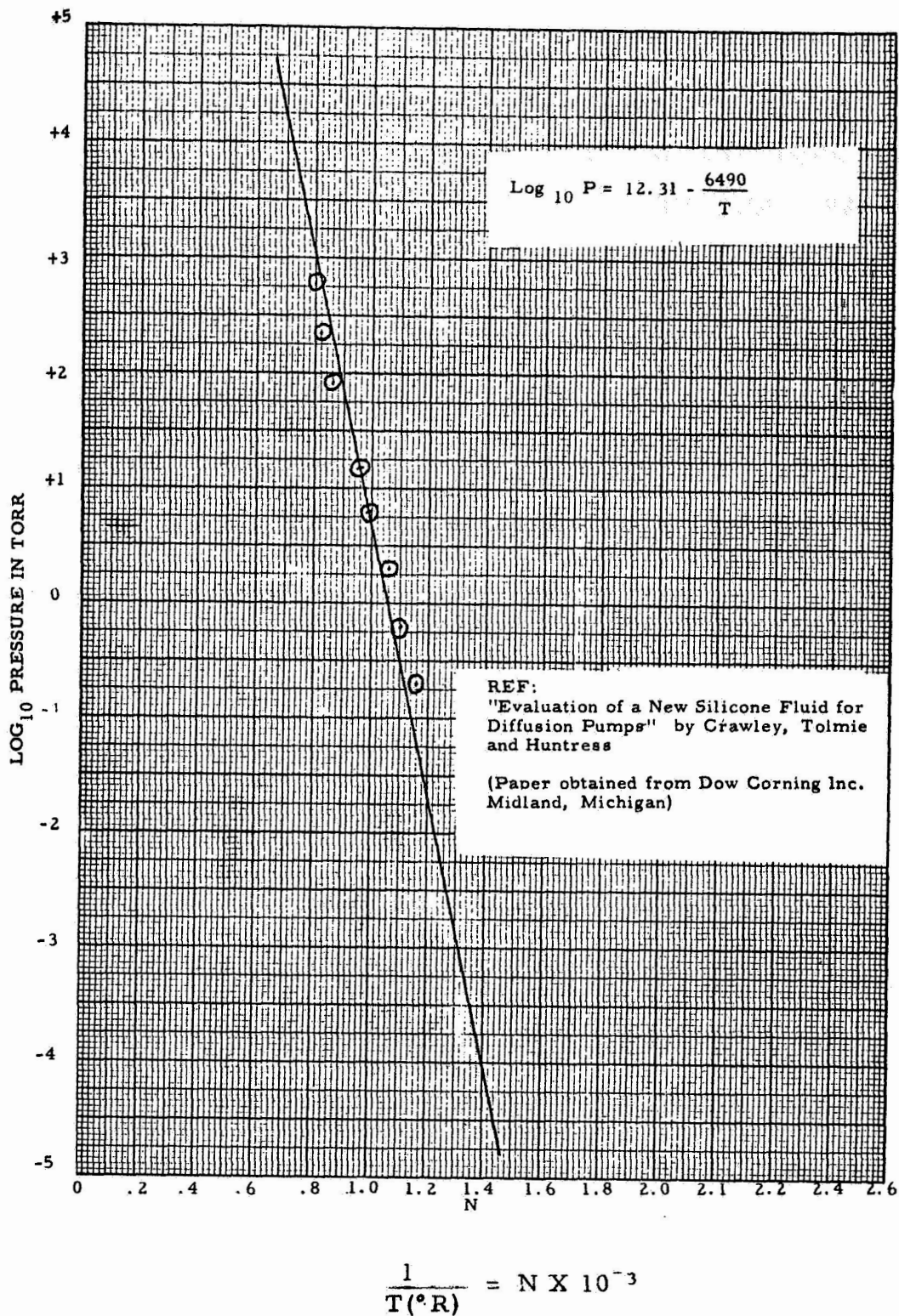


Fig. 1

VAPOR PRESSURE OF DC-705

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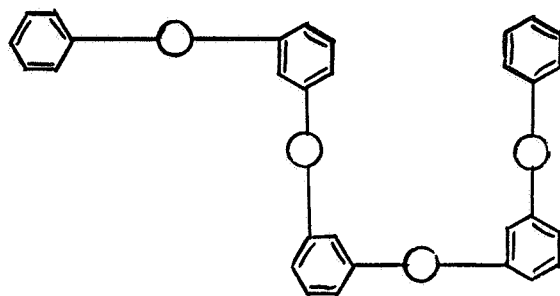
105

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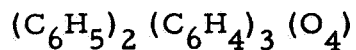
DIFFUSION PUMP OIL: Convalex 10 (PURIFIED OS-124)

MOLECULAR STRUCTURE



 = PHENYL GROUP

FORMULA

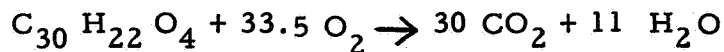


MOLECULAR WEIGHT

446.5

TECHNICAL NAME

STOICHIOMETRIC FORMULA



ISOTHERMAL PRESSURE RATIO

$$\frac{P_2}{P_1} = \frac{41}{34.5} = 1.19$$

THERMAL STABILITY

Decomposition begins at 847°F (isoteniscope)

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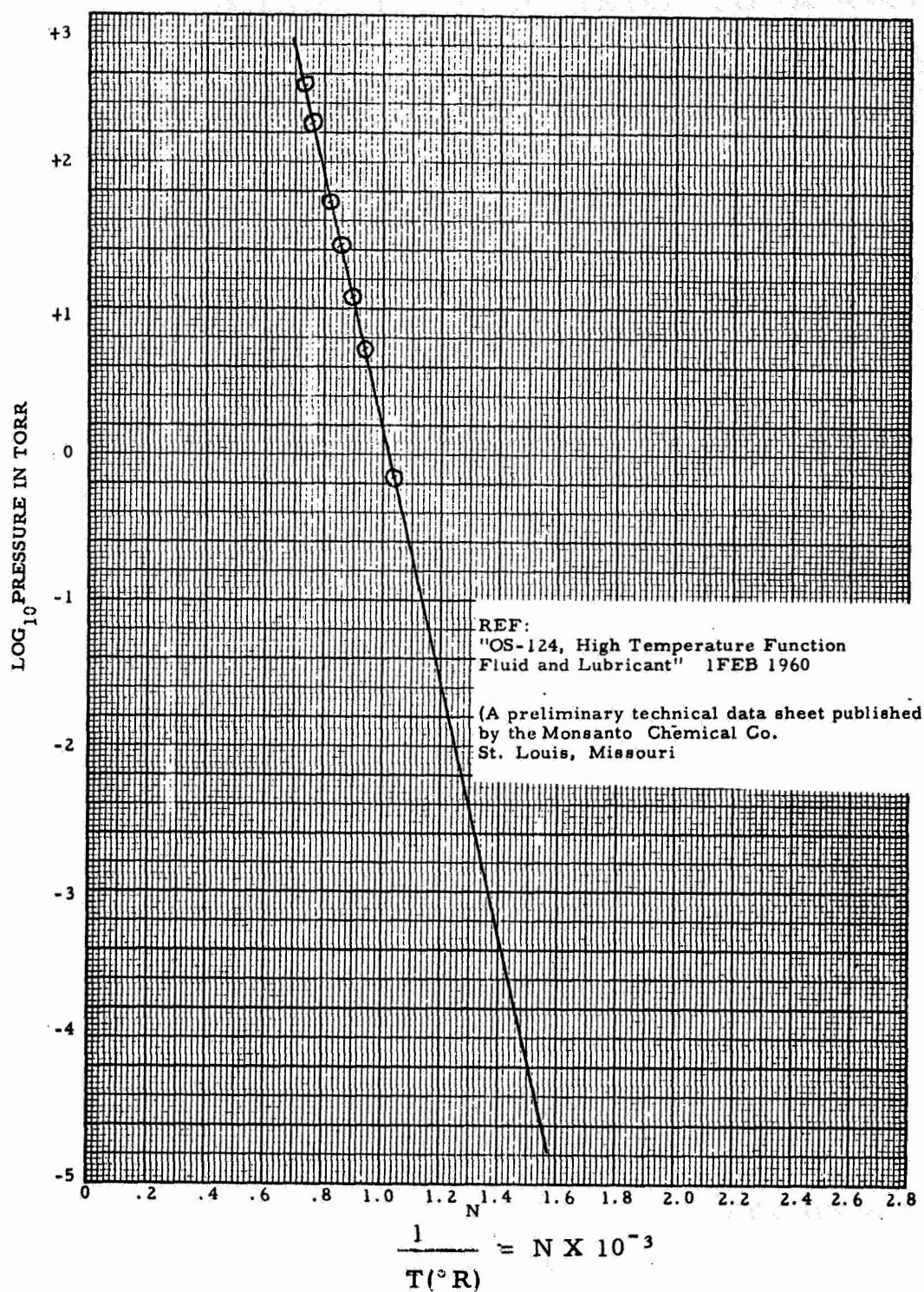


Fig. 2

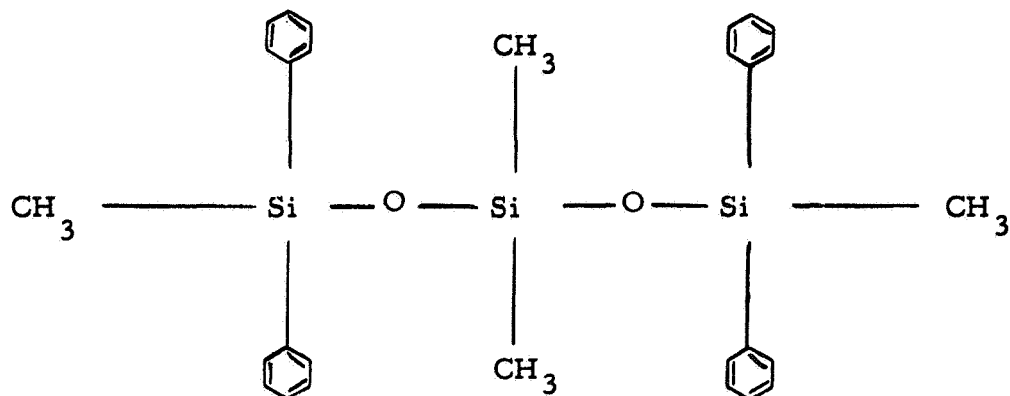
VAPOR PRESSURE OF CONVALEX 10


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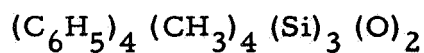
DIFFUSION PUMP OIL; DC 704

MOLECULAR STRUCTURE



 = PHENYL
GROUP

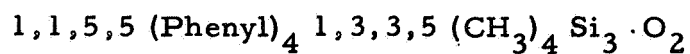
FORMULA



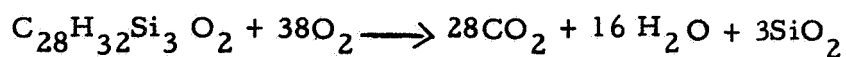
MOLECULAR WEIGHT

484.7

TECHNICAL NAME



STOICHIOMETRIC FORMULA



SiO_2 is a solid

ISOTHERMAL PRESSURE RATIO

$$\frac{P_2}{P_1} = \frac{44}{39} = 1.13$$

THERMAL STABILITY

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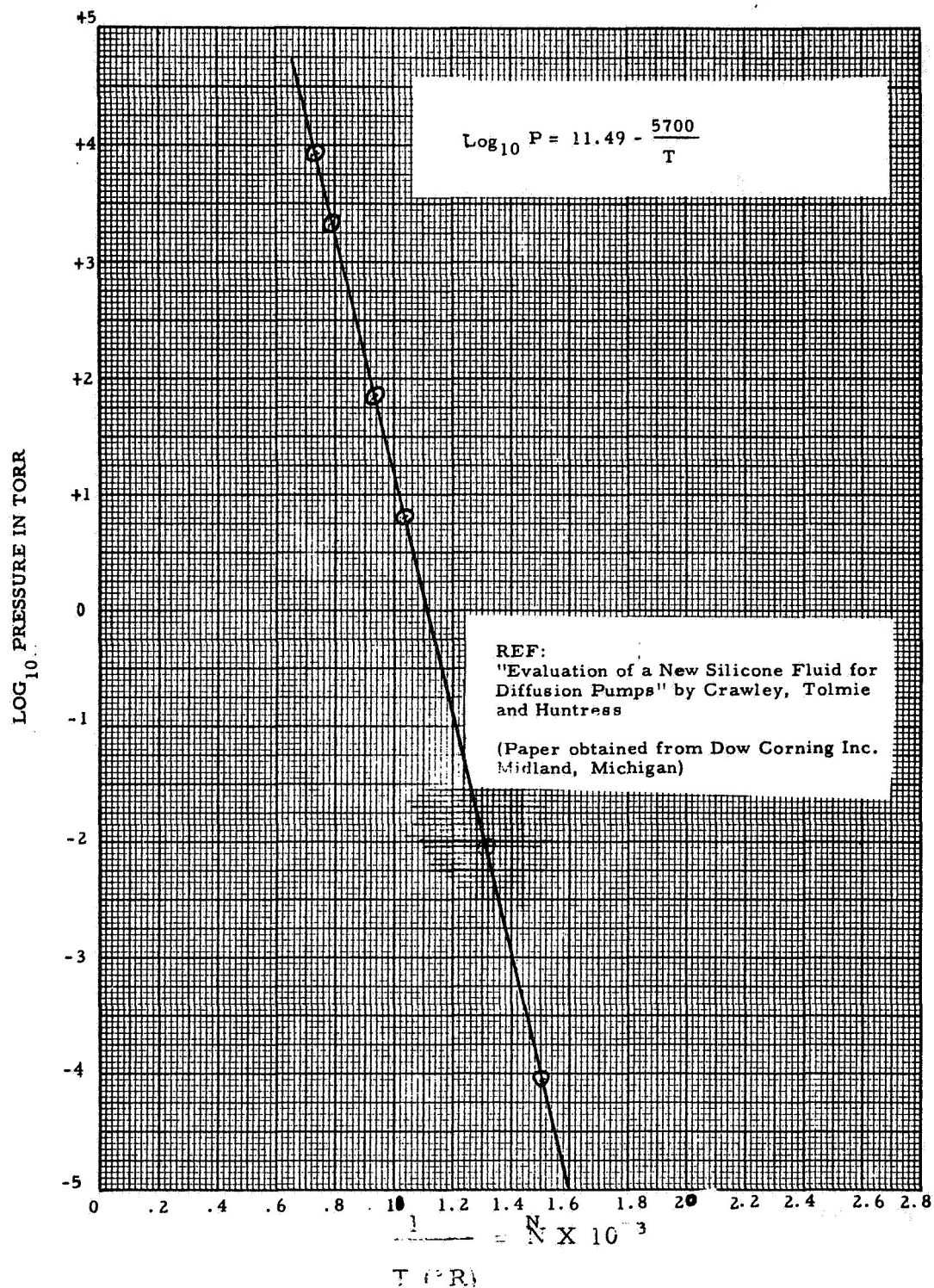


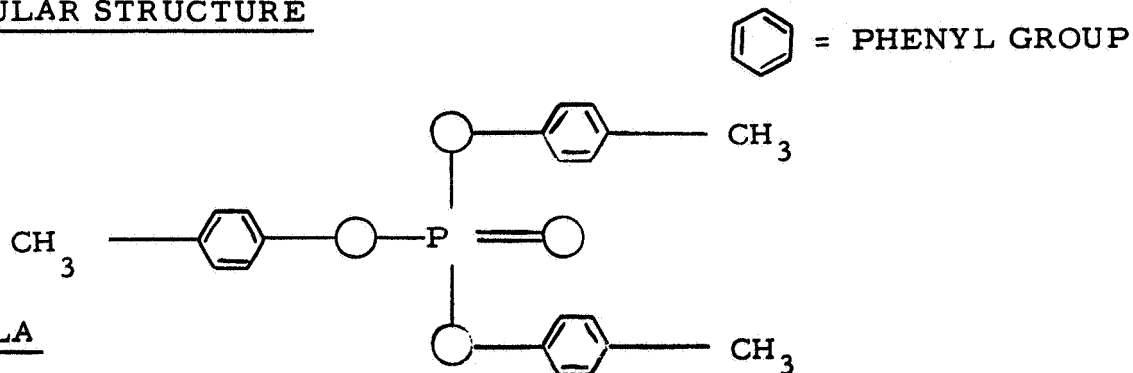
Fig. 3

VAPOR PRESSURE OF DC-704

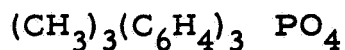
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DIFFUSION PUMP OIL: Tricresyl Phosphate

MOLECULAR STRUCTURE



FORMULA



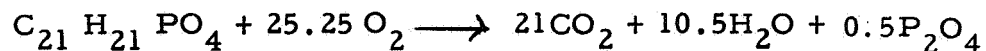
MOLECULAR WEIGHT

368.4

TECHNICAL NAME

O-Tolyl Phosphate

STOICHIOMETRIC FORMULA

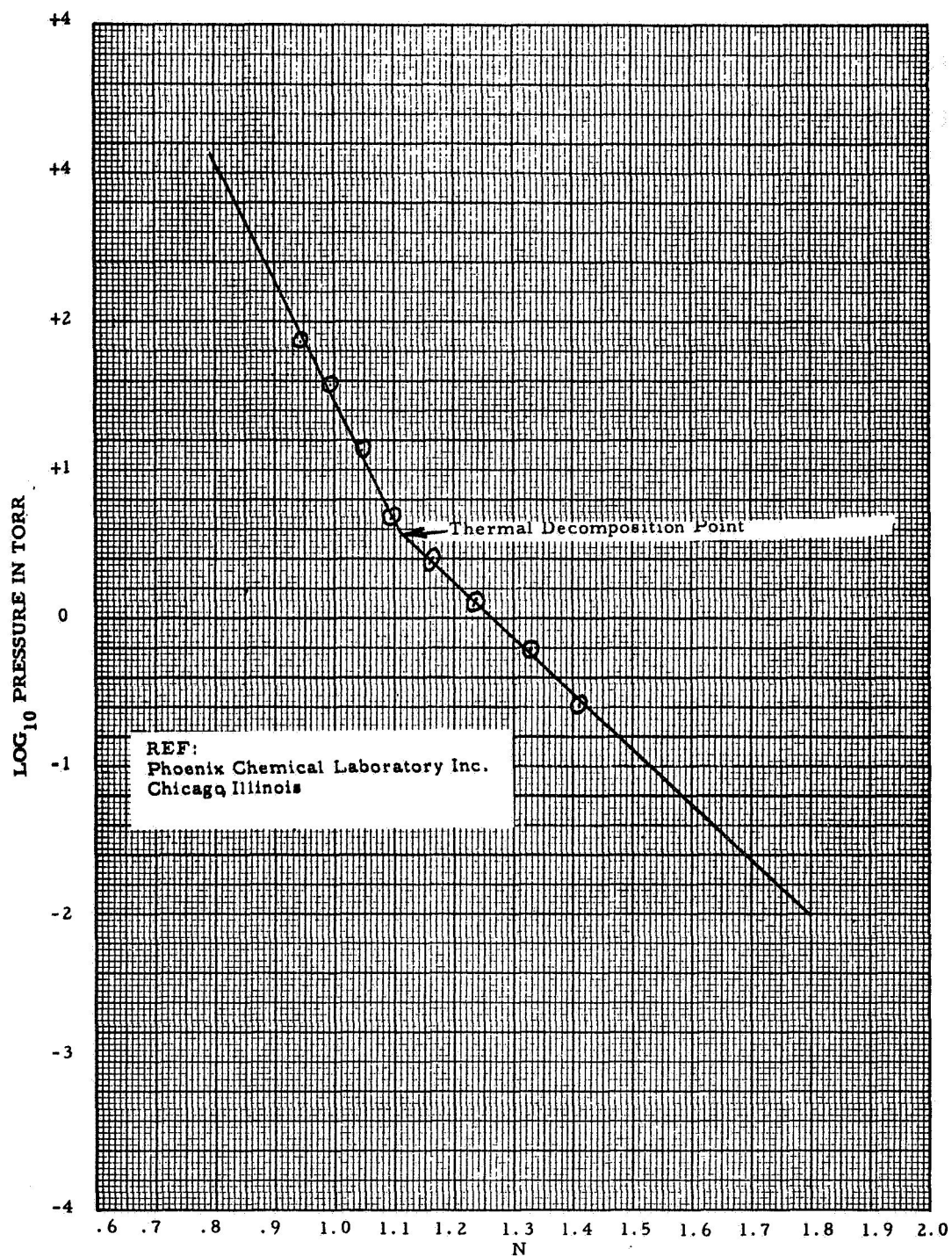


ISOTHERMAL PRESSURE RATIO

$$\frac{P_2}{P_1} = \frac{32.00}{26.25} = 1.215$$

THERMAL STABILITY

Decomposition begins to occur at 430°F (isoteniscope)



$$\frac{1}{T (^{\circ}\text{R})} = N \times 10^{-3}$$

Fig. 4 VAPOR PRESSURE OF TRICRESYL PHOSPHATE

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DIFFUSION PUMP OIL: Convoil 20

MOLECULAR STRUCTURE

FORMULA

$C_{29.5} H_{45.1}$ (an average) This formula was deduced from the experimental determination of the carbon

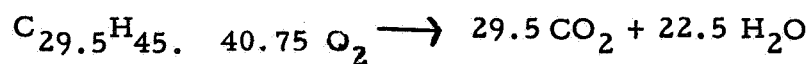
MOLECULAR WEIGHT

400

hydrogen ratio at Armour and the reported molecular weight.

TECHNICAL NAME

STOICHIOMETRIC FORMULA



ISOTHERMAL PRESSURE RATIO

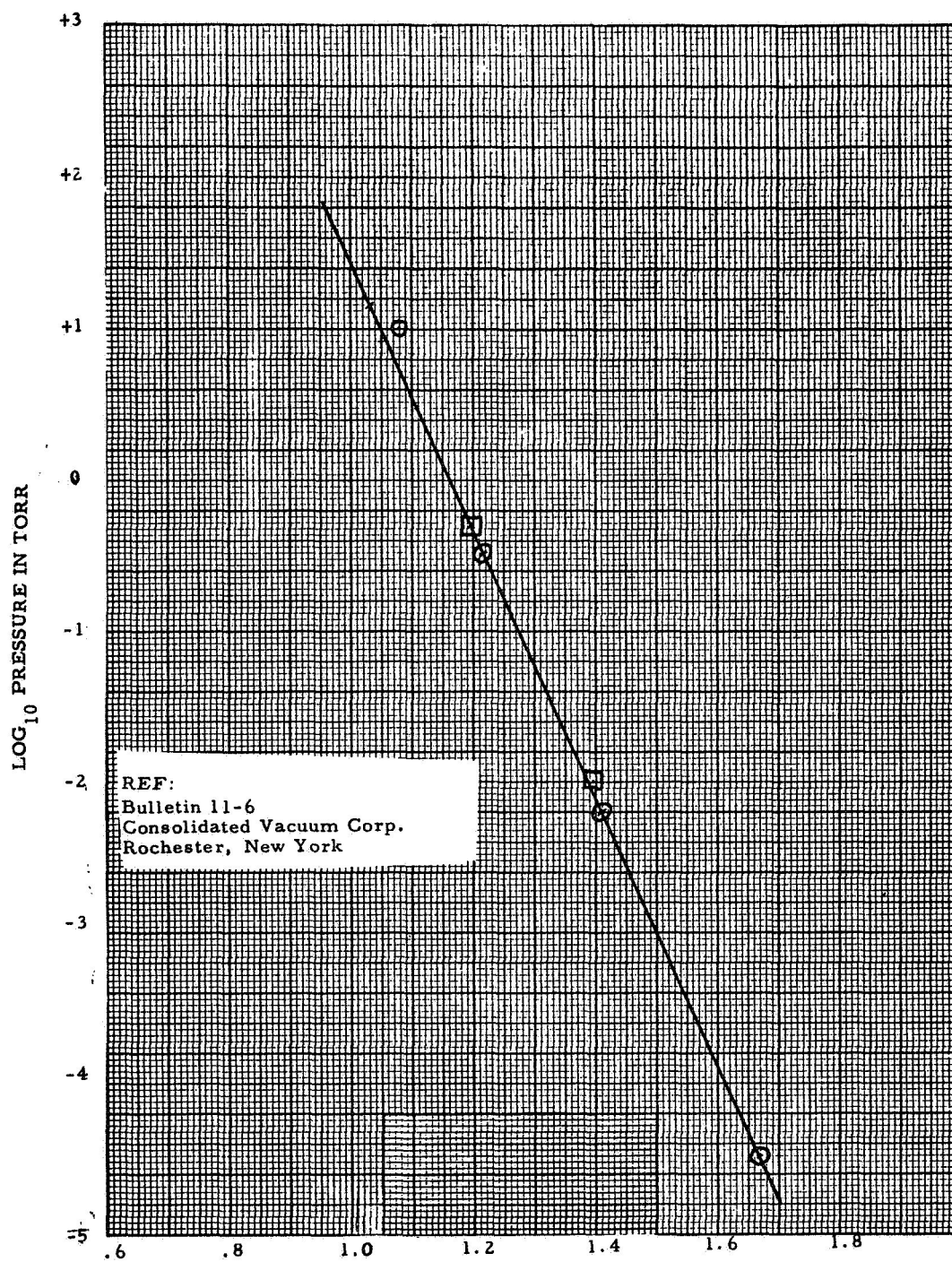
$$\frac{P_2}{P_1} = \frac{52.00}{41.75} = 1.242$$

THERMAL STABILITY

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$$\frac{1}{T (^{\circ}R)} = N \times 10^{-3}$$

Fig.5 VAPOR PRESSURE FOR CONVOIL 20.

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APPENDIX D

SUMMARY OF SPARK IGNITION INVESTIGATION BY LEWIS AND VON ELBE

The investigators, Blanc, M. V., Guest, P. G., von Elbe, Guenther, and Lewis, have done a large amount of work in determining minimum ignition energies and spark gap for gaseous mixtures. For their investigation, it was necessary to develop an apparatus which could dissipate a measurable amount of energy into the gas mixture. Several difficult problems are associated in the design of such an apparatus. The final design which was used is described briefly in this section and is shown schematically in Fig. 1. All parts of the system were designed to minimize resistance and corona losses. Freedom from dielectric hysteresis was assured by using air capacitors. The capacity was variable between 100 to 5000 $\mu\mu$ fd. and its exact value was determined by a Wien bridge. A resistor rod of the order of 10^{11} ohms was used to allow slow charging of the capacitor. The resistance necessary to charge a given capacitor in a given time to a specified voltage knowing the source voltage may be found by considering the integral equation for current flow in a R-C circuit.

$$iR + \frac{1}{C} \int i dt = E$$

where i = current, R = Resistance, C = capacitance, E = source voltage and t = time. For a system which is initially uncharged.

The solution of this equation is

$$i = \frac{E}{R} e^{-t/RC}$$

and is found by substituting $i = \frac{dq}{dt}$ and solving the resulting differential equation for q .

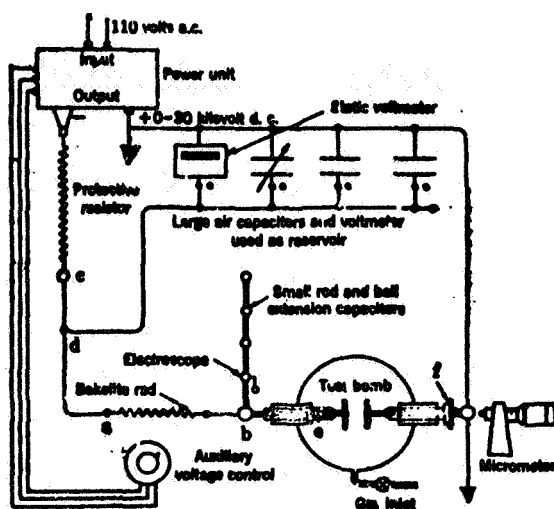


Fig. 1 APPARATUS ARRANGED FOR SPARK CIRCUITS
OF VERY LOW CAPACITANCE (BLANC GUEST,
VON ELBE, AND LEWIS)

The voltage V in the capacitor at any time is given by;

$$V = E (1 - e^{-t/RC})$$

The resistance may be found by substituting the known quantities into this equation. For example, the resistance required for a charging time of 30 seconds, 10,000 volts initial, 8000 volts final, and a capacity of 200 μ fd is approximately 10^{11} ohms.

The capacitor discharge occurs automatically in this set up since the capacitor voltage will increase until the voltage is high enough for breakdown. The energy dissipated may be calculated by the equation $\frac{1}{2} CV^2$. Calorimetric measurements of the heat generated by sparks have shown that immediately after a spark discharge, 95% of $\frac{1}{2} CV^2$ is available in the gas in the form of heat. If this does not cause the mixture to ignite, a larger capacitance is used and the process is repeated. Sparking occurred after a statistical time lag which was inconveniently long and in general it was possible to reduce the lag substantially by placing radium capsules of various strengths in the bomb.

By moistening the resistor rod slightly with glycerin or another liquid, or conversely, by rubbing the rod with a dry cloth, the rate of leakage could be accelerated or retarded at will. The capacitance of the spark circuit could be adjusted by means of small extension capacitors, as shown in Fig. 1, to any value from about 100 μ fd to the capacitance of the electrode itself. The smallest capacitance was about 1 μ fd. A bakelite rod could be inserted in the circuit for a high resistance leakage connection with the capacitors to the electrode.

Experiments were made with mixtures of oxygen and various inert gases with pure hydrocarbons, hydrogen, and other compounds. Two typical curves obtained are shown in Fig. 2.

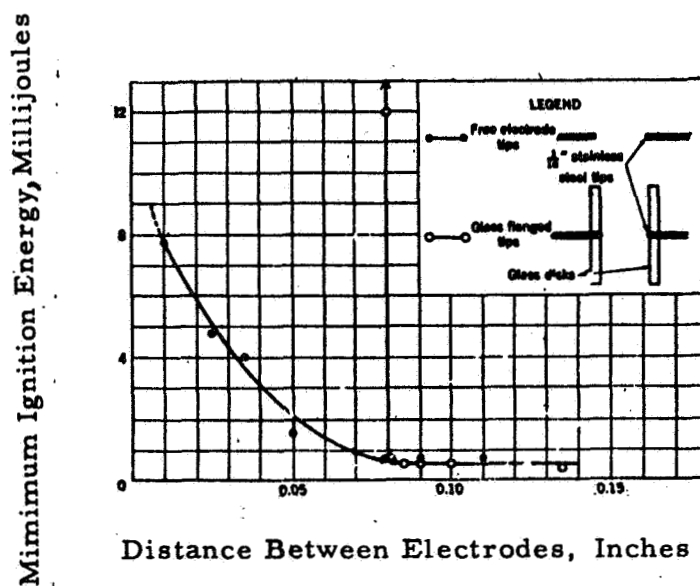


Fig. 2 MINIMUM IGNITION ENERGIES FOR FREE AND GLASS-FLANGED ELECTRODE TIPS AS FUNCTION OF ELECTRODE DISTANCE. STOICHIOMETRIC MIXTURE OF NATURAL GAS (ABOUT 83% CH₄ + 17% C₂H₆) AND AIR AT 1 ATMOSPHERE PRESSURE

One curve corresponds to a series of experiments in which the electrode terminals were tipped with glass flanges. The other curve corresponds to a series of experiments in which the electrodes were not tipped.

Note that below a spacing of 0.08 inches, the energy required for ignition increases very quickly for the untipped and increases to infinity for the tipped electrode. This is caused by the quenching effect of the electrodes.

Fig. 3 shows the effect of pressure upon the minimum ignition energy. Note that the minimum ignition energy is constant for a portion of each of these curves. As a matter of fact, a spacing of 0.35 inches will suffice to enable ignition of all these gas mixtures at or very near the minimum ignition energy for all pressure from 0.2 atmospheres to 0.67 atmospheres. This indicates that one spacing may be used for several different pressures and that only the minimum ignition energy will be expended in ignition.

Table 1 shows that the minimum ignition energy is independent of the voltage drop across the electrodes.

A number of tests in which a helix of heavy wire added to the circuit failed to show any effect of moderate changes of inductance and/or oscillatory frequency in the value of the minimum ignition energy.

In some experiments, a switch was used instead of the bakelite rod. There is possibly an energy loss in this switch, but because of the large time lag needed for a spark to occur, this effect was considered non-existent.

In Fig. 4, a representative curve of minimum ignition energy distance between plate electrodes has been extended to note the effect of very large spark energies. It is seen that the quenching distance does not diminish when very large sparks are used, but on the contrary increases.

Fig. 5 shows families of curves of minimum ignition energies for various mixture compositions and pressures. An important effect of inert gas dilution is noted. For example, near stoichiometric composition, the minimum ignition energy is found to be 100 times larger for air than for oxygen.

TABLE 1
IGNITION ENERGY AS A FUNCTION OF BREAKDOWN VOLTAGE

Experimental Conditions	Gap Voltage, Kilovolts	Minimum Ignition Energy Millijoules*
8.7 percent natural gas** in air at 1 Atmosphere	6.1	0.5
	9.9	0.5
0.5 Atmosphere	8.6	1.7
	10.2	1.7
0.33 Atmosphere	5.4	2.4
	7.5	2.4
0.25 Atmosphere	4.5	4.0
	6.8	4.2
8.5 percent methane in air at 0.33 Atmosphere	14.2	2.6
	20.0	2.6

*The distance between the electrodes was held constant for each pair of runs.

**Approximately 83 percent CH_4 and 17 percent C_2H_4 .

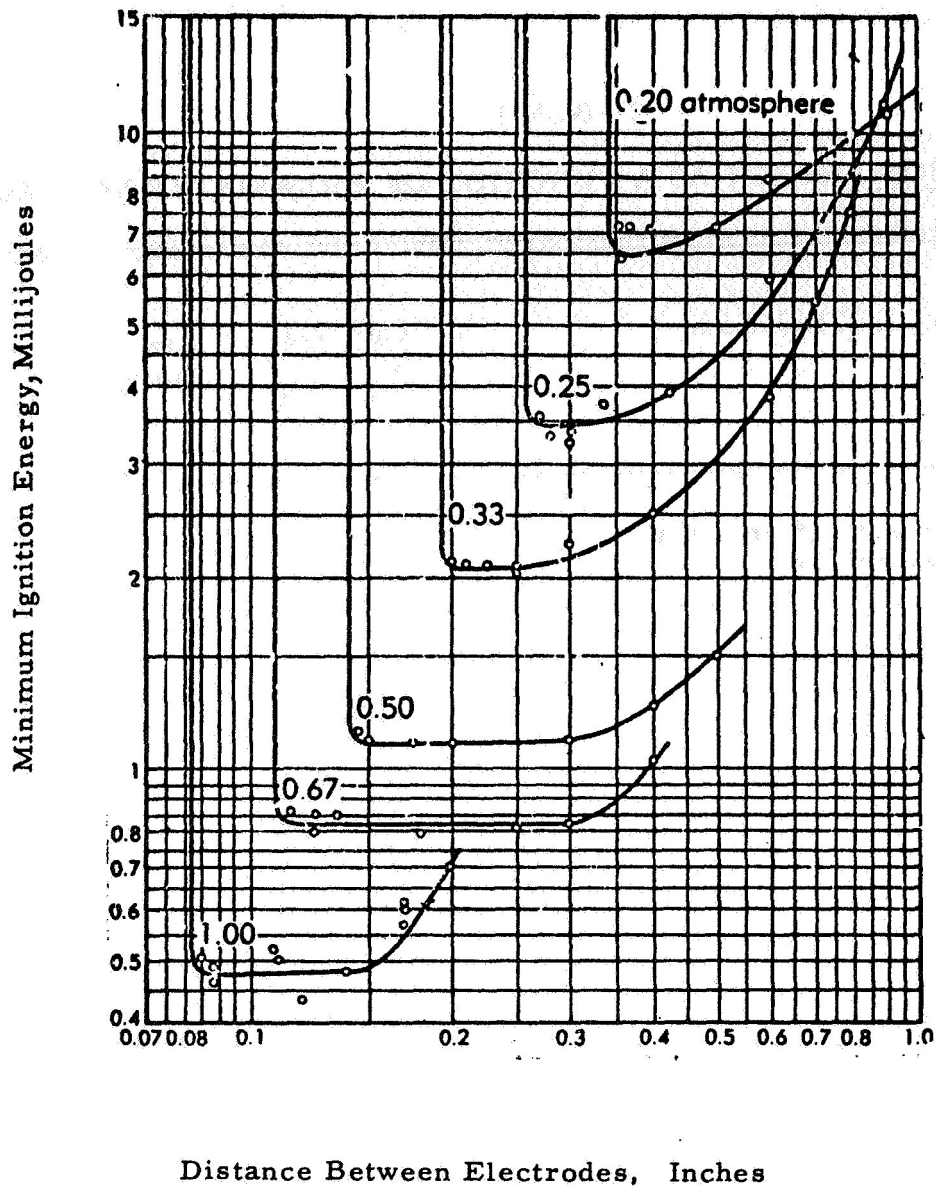


Fig. 3 MINIMUM IGNITION ENERGIES FOR GLASS-FLANGED ELECTRODE TIPS AS FUNCTIONS OF ELECTRODE DISTANCE AND PRESSURE. STOICHIOMETRIC MIXTURE OF METHANE AND
AIR

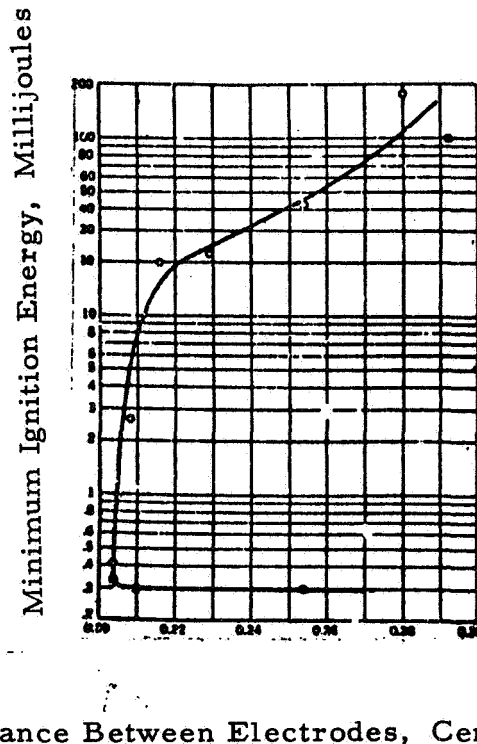


Fig. 4 INCREASE OF QUENCHING DISTANCE AT LARGE SPARK ENERGIES. 8.5% MIXTURE OF METHANE AND AIR AT ONE ATMOSPHERE

The circuit employed in this project (Fig. 7 of Appendix B) is a simplified form of Fig. 1. Whenever possible, the information of this reported investigation was used to design our equipment.

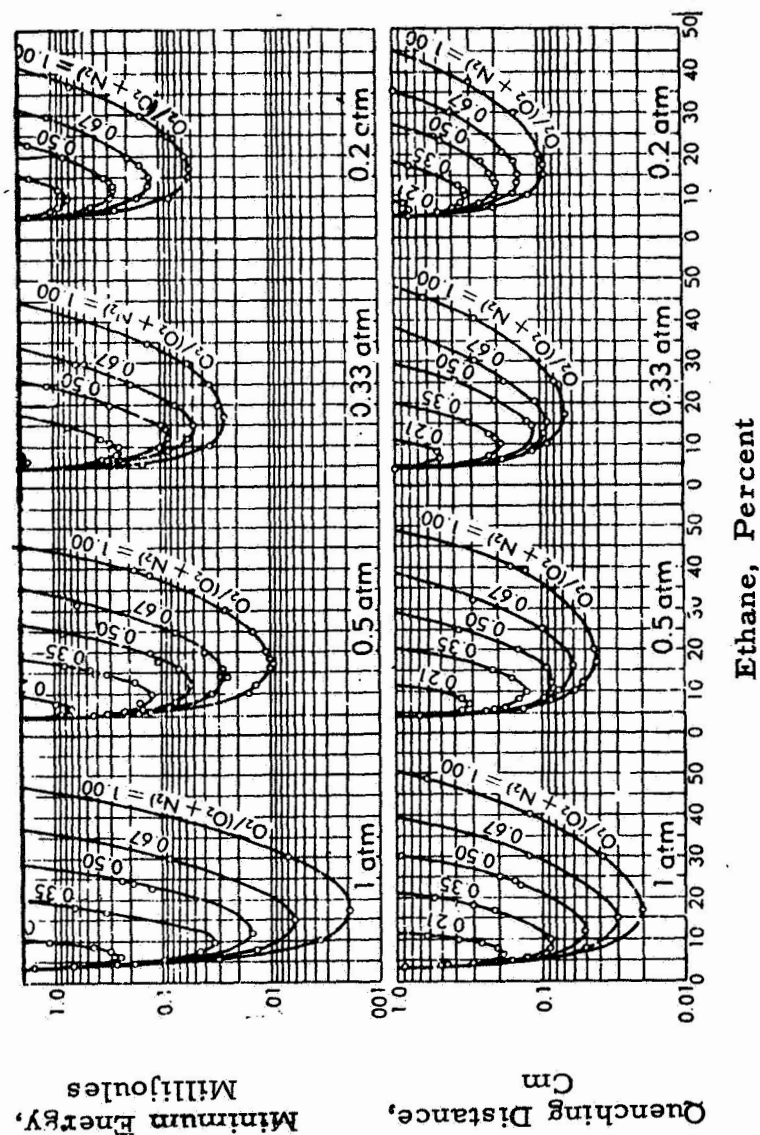


Fig. 5

MINIMUM SPARK IGNITION ENERGIES IN MILLIJOULES OF MIXTURES OF ETHANE, OXYGEN, AND NITROGEN AT ONE ATMOSPHERE PRESSURE AND LOWER, AND QUENCHING DISTANCES BETWEEN FLANGED ELECTRODES FOR THE SAME MIXTURES. CURVES CORRESPOND TO CONSTANT RATIOS OF OXYGEN AND NITROGEN